CH2M HILL
18 Tremont Street
Suite 700
Boston, MA 02108
Tel 617.523.2260
Fax 617.723.9036

December 4, 2013

Mr. Richard Ho, RPM/OSC
U.S. Environmental Protection Agency, Region 2
Emergency and Remedial Response Division
290 Broadway, Floor 19
New York, NY 10007-1866

Subject: Quanta Resources Corporation Superfund Site, Operable Unit 1 (OU1) Vapor Intrusion—Work Plan for the Winter 2013/2014 Monitoring Events at 115 River Road, 163 Old River Road, and 103 River Road

Dear Mr. Ho:

CH2M HILL has prepared this letter Work Plan on behalf of Honeywell International Inc. (Honeywell) to present the sampling methods and procedures for the winter 2013/2014 vapor intrusion (VI) monitoring events at the 115 River Road, 163 Old River Road, and 103 River Road buildings, which are associated with the Quanta Resources Corporation Superfund Site in Edgewater, New Jersey (the Site). These monitoring events are being conducted as part of a routine monitoring effort being performed at the Site until the final remedy has been implemented in accordance with the Record of Decision (ROD) and as detailed during the July 23, 2013 meeting between the New Jersey Department of Environmental Protection (NJDEP), the U.S. Environmental Protection Agency (USEPA), and Honeywell. The objective of this monitoring is to confirm that the conditions at each building are similar to those previously documented in the 2011 ROD; specifically, the ROD states on Page 29: although "…elevated levels of site contaminants…" were detected in subslab soil gas, "…the detected levels [in indoor air] have not exceeded [US]EPA's guidelines for exposure to indoor air."

The scope proposed in this Work Plan covers a single sampling event that will involve the collection of subslab soil gas, crawl space, and outdoor and indoor air samples and subsequent reporting. Future monitoring events will be detailed within the Basis of Design Report to be submitted to USEPA in 2014.

USEPA and NJDEP comments on the draft version of this work plan have been incorporated into this final version. The comments and responses are provided in Attachment A.

Background

VI investigation activities at the Site began in 2006 as part of the OU1 remedial investigation, and VI investigation and monitoring has continued annually since then at the 115 River Road, 163 Old River Road, and 103 River Road buildings. The investigation and monitoring activities were conducted in accordance with work plans approved by the USEPA and the NJDEP. The ROD and Administrative Order of Consent for OU1 summarized the conclusions of the VI investigation and identified continuing activities for addressing VI during implementation of remedial action at the Site. As detailed within the ROD, results of the VI investigation and monitoring events since 2006 indicate that the VI pathway has not caused indoor air concentrations to exceed USEPA's guidelines for exposure to indoor air.

Work plans were submitted to USEPA in December 2012 to define the specific long-term monitoring (LTM) activities in the three buildings during remedial action at OU1. LTM activities identified in these plans included: annual monitoring at the 115 River Road building until it underwent demolition as required to perform remedial action; monitoring conducted every 5 years at the restaurant building; and discontinuing monitoring at the Medical Arts building unless changing conditions warranted resumed monitoring.

Following agency comments submitted in February 2013, discussions of LTM approaches were deferred and work plans were submitted to the agencies describing monitoring activities only for 2013, which were conducted in March 2013 and detailed in the letter report *Quanta Resources Corporation Superfund Site, Operable Unit 1 (OU1) Vapor Intrusion—Results of the 2013 Monitoring Events at 115 River Road, 163 Old River Road, and 103 River Road,* dated May 21, 2013 (CH2M HILL, 2013b).

Honeywell met with the agencies on July 23, 2013 to review VI investigation and monitoring results for the Site, and to discuss a path forward for VI activities during OU1 remedial design and subsequent remedial action. The outcomes from that meeting are:

- Submitting this letter Work Plan for winter 2013/2014 VI monitoring at the three buildings
- Performing that monitoring during the fourth quarter of 2013
- Reporting the results to USEPA and NJDEP

This Work Plan incorporates responses to NJDEP comments received from USEPA on July 23, 2013, via e-mail (Attachment A). The results collected during this monitoring event will be reviewed to define LTM needed to support remedial action at OU1. OU1 remedial action is scheduled to begin in late 2014/early 2015. It is anticipated that LTM VI monitoring and ongoing air monitoring during remediation will be incorporated into the Remedial Action Work Plan for OU1.

The proposed monitoring locations in this Work Plan reflect the comments provided by NJDEP.

115 River Road (Office/Commercial Use Building)

The main building at 115 River Road is an old manufacturing building that has been converted into 11 different tenant-occupied “buildings,” which are further divided into multiple commercial/office spaces. Some portions of the building are constructed on top of a crawl space, while others are either slab-on-grade or have a basement, as shown in Figure 1. There is an interim VI mitigation measure in place in the Building 7/8 basement and Building 6 crawl space; it consists of a ventilation system that generates approximately 15 indoor-to-outdoor air exchanges per hour. The system is currently operated by the building owner, who keeps it running 24 hours per day. The building owner has also performs housekeeping activities within Building 7/8 (for example, keeping covers on the sumps and floor drains) to reduce the potential for VI. CH2M HILL conducts monthly inspections of the ventilation system and basement.

163 Old River Road Building (Tomaso’s Restaurant)

The 163 Old River Road building had multiple former uses (for example, quality control laboratory, storage, and office space) but has since been converted into a restaurant. The building is currently occupied by Tomaso’s (previously Jono’s) Restaurant. The building is approximately 6,000 square feet (ft^2) and consists of two stories with a single-story front dining area.

103 River Road Building (Medical Arts)

The 103 River Road building was constructed in the early 1980s and is two-and-a-half stories, occupying a footprint of approximately 5,000 ft^2 (100 feet long by 50 feet wide). The building contains three separate office spaces: a medical office and a dental office on the first floor, and a cardiologist office on the second floor.

Regulatory Framework

The VI monitoring events and evaluations are performed in accordance with the following USEPA-approved work plans and Quality Assurance Project Plan, and guidance documents:

- CH2M HILL, *Quality Assurance Project Plan, Quanta Resources Corporation Superfund Site, Operable Unit 1, Edgewater, New Jersey*, August 2012a.

- CH2M HILL, *Quality Assurance Project Plan Addendum, Quanta Resources Corporation Superfund Site, Operable Unit 1, Edgewater, New Jersey*, August 2012b.
- USEPA, *OSWER Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils* (Subsurface Vapor Intrusion Guidance), 2002
- USEPA Office of Superfund Remediation and Technology Innovation (OSRTI), Vapor Intrusion Screening Level Calculator Tool, version 3.0, May 2013, using the May 2013 Regional Screening Levels
- NJDEP, *Vapor Intrusion Technical Guidance* (VIT Guidance) and the associated NJDEP Vapor Intrusion Screening Level Tables, March 2013
- Interstate Technology and Regulatory Council (ITRC), *Vapor Intrusion Pathway: A Practical Guideline*, 2007

When there is disparity between USEPA and NJDEP guidance, the USEPA guidance or USEPA Region 2 standard practices will take precedence because USEPA Region 2 is the lead regulatory agency for the Site, in accordance with the agency-approved work plans.

Field Sampling Plan

Sampling activities will be performed in accordance with the previous USEPA-approved VI monitoring work plans with the appropriate standard operating procedures (SOPs) provided in Attachment B and the updated Uniform Federal Policy–Quality Assurance Project Plan (UFP-QAPP; Attachment C), with the exception of the deviations detailed in this section. Field data will be recorded in the field data forms provided in these SOPs. Field activities will be conducted in accordance with the project *Health and Safety Plan* (HASP) (CH2M HILL, 2013a).

A building survey will be performed at each building at least a week prior to indoor air sampling activities. The Indoor Air Building Survey and Sampling Form, which is Appendix D of the NJDEP (2013) VIT Guidance, will be completed during the building survey.

Sampling at 115 and 103 River Road will be performed during the work week (Monday to Friday) while tenants are occupying the building. Sampling at 163 Old River Road will be performed while the restaurant is closed, sometime between 9 pm Sunday and 5 pm Tuesday, as requested by the building owners and consistent with previous events. The heating, ventilation, and air conditioning (HVAC) system will be set to typical operating conditions, and the building's windows and doors will be kept shut during the sampling event at each building. The field sampling team will check on each building periodically to verify these conditions persist throughout the entire sampling period.

Sampling will not be performed during storm events or within 48 hours of a significant rain event (that is, greater than 1 inch of rain in a 24-hour period) because of the potential influence such conditions may have on indoor air, outdoor air, and subslab soil gas. Information on weather conditions in Edgewater, New Jersey during the sampling event will be obtained from Weather Underground's Web site.[1]

Sampling Locations

The sampling locations are provided in Tables 1a, 1b, and 1c, shown in the figures attached, and can be summarized as follows:

- **115 River Road.** The sampling locations were modified from previous years to address comments from NJDEP during the July 2013 and via e-mail (Attachment A) and to address additional comments from the NJDEP on this draft Work Plan (response to comments provided as Attachment D). Indoor air sampling will be performed at each of the occupied tenant spaces on the first floor at the 115 River Road building. Indoor

[1] http://www.wunderground.com/.

air samples will also be collected from the unoccupied Building 7/8 basement where the ventilation system is operating and unoccupied the Building 10 basement. Additionally, indoor air samples will be collected from the second and third floor occupied tenant spaces closest to the elevator in Building 8 to assess if the elevator is acting as a preferential vapor pathway and one sample will be collected within the Building 3 second floor occupied space. Crawl space air samples will be collected at Buildings 2, 3, 4, and 6.

- **163 Old River Road.** The same locations that were sampled in previous years will be sampled during the winter 2013/2014 VI monitoring event.
- **103 River Road.** The same locations that were sampled in previous years will be sampled during the winter 2013/2014 VI monitoring event. The three existing subslab soil gas probes will be removed and Cox Colvin vapor pins will be installed in the similar location due to two of the existing subslab soil gas probes being rusted shut.

Sampling Methods

The samples (whatever combination of indoor, outdoor, or crawl space air, or subslab soil gas samples are proposed) will be collected concurrently over a 24-hour period at each building. The samples will be collected in 6-liter (L) Summa canisters equipped with flow controllers. Each Summa canister will be placed at the sampling location, turned on, and left undisturbed for 24 hours. The Summa canisters will be checked after 20 hours to make sure that canister pressure does not reach zero. The indoor air samples in occupied spaces and the outdoor air samples will be placed at a height of approximately 3 to 5 feet above the ground (roughly breathing zone height).

The three existing subslab soil gas probes at the 103 River Road building will be removed, and Cox Colvin brand vapor pins will be installed after re-drilling those holes. The vapor pins will be installed, leak checked, and sampled in accordance with the Cox Colvin and CH2M HILL SOPs in Attachment B.

The subslab soil gas probes at the 163 Old River Road will be leak checked, purged, and sampled in accordance with the CH2M HILL SOP in Attachment B.

Sample Analysis

The Summa canisters will be managed and shipped to the analytical laboratory, ALS Environmental (ALS) (formerly Columbia Analytical Services) in Simi Valley, California, under chain-of-custody procedures. The indoor, crawl space, and outdoor air, and subslab soil gas samples will be analyzed for a shortened volatile organic compound (VOC) analyte list by USEPA Method TO-15. The air samples will be analyzed using the TO-15 Selective Ion Mode (SIM) for naphthalene to achieve lower reporting limits. ALS is certified for TO-15 analyses by NJDEP (NJ Certification No. CA009). Ten-day turnaround times will be requested from ALS.

The shortened VOC analyte lists that will be analyzed for each building are shown in Tables 2 through 4.

Data Evaluation and Reporting

The project chemist will perform a data quality evaluation upon receipt of the analytical data from ALS to determine if the data quality objectives are met.

A letter report will be prepared to summarize the results of the winter 2013/2014 VI monitoring at the three buildings. The letter will be similar to what was provided for the winter 2012/2013 VI monitoring events and will include a brief summary of the results of the sampling events at each building, with figures illustrating the sampling locations, and data tables showing the analytical results compared to the USEPA and NJDEP screening levels presented in Tables 3a, 3b, 4a, and 4b. Attachments providing the sampling logs, building survey forms, data quality evaluation, historical data tables, and figures showing shallow groundwater VOC concentrations near each building will also be included.

Additional Data Submittals

Four submittals of the validated analytical data will be prepared and transmitted to USEPA and NJDEP in accordance with the NJDEP (2013) VIT Guidance as follows:

1. The indoor air sample data will be compared to the NJDEP (2013) Rapid Action Levels presented in Table 2 in Attachment B, and the results will be submitted via email to USEPA and NJDEP within 14 to 30 days (depending on the results) of receiving validated results.
2. A compact disc (CD) of the full laboratory data packages, including the unit conversion tables on a CD and a hard copy of the completed Full Laboratory Data Deliverables Form, will be submitted via certified mail to NJDEP within 14 to 30 days (depending on the results) of receiving validated results.
3. Portable document format (PDF) files of the indoor and outdoor air sample data with sample location keys and figures will be submitted to the New Jersey Department of Health via email within 14 days of receiving validated results.
4. Letters providing and explaining the sample results will be submitted via certified mail to the building owners, with copies to the local health department, NJDEP, and USEPA within 14 to 30 days (depending on the results) of receiving validated results.

References

CH2M HILL. 2012a. *Quality Assurance Project Plan, Quanta Resources Corporation Superfund Site, Operable Unit 1, Edgewater, New Jersey*. August.

CH2M HILL. 2012b. *Quality Assurance Project Plan Addendum, Quanta Resources Corporation Superfund Site, Operable Unit 1, Edgewater, New Jersey*. August.

CH2M HILL. 2013a. *Health and Safety Plan*.

CH2M HILL. 2013b. *Quanta Resources Corporation Superfund Site, Operable Unit 1 (OU1) Vapor Intrusion—Results of the 2013 Monitoring Events at 115 River Road, 163 Old River Road, and 103 River Road*. May 21.

Interstate Technology and Regulatory Council (ITRC). 2007. *Vapor Intrusion Pathway: A Practical Guideline*.

New Jersey Department of Environmental Protection (NJDEP). 2013. *Vapor Intrusion Technical Guidance and the associated NJDEP Vapor Intrusion Screening Level Tables*. March.

U.S. Environmental Protection Agency (USEPA). 2002. *OSWER Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils*.

U.S. Environmental Protection Agency (USEPA). 2013. Office of Superfund Remediation and Technology Innovation (OSRTI), Vapor Intrusion Screening Level Calculator Tool. Version 3.0. May.

Please feel free to contact me at 267.250.7387 or Steve Coladonato, Honeywell Remediation Manager, at 302.791.6738, if you have any questions or comments regarding the Quanta project.

Sincerely,

CH2M HILL

Stephen J. Zarlinski
Project Manager

cc:	Clay Monroe/USEPA	Steve Coladonato/Honeywell
	Erica Bergman/NJDEP	Helen Fahy/Fahy Associates

ES091313103509BOS

Tables

TABLE 1A

Proposed Sample Locations - Winter 2013/2014 Vapor Intrusion Monitoring Event

115 River Road Building

Quanta Site, Edgewater, New Jersey

Indoor Air Sample Locations

Location ID	Bldg #	Floor	Sample Location Description
Q1-IA-32	2	1st	Main open space
Q1-IA-13	3	2nd	Occupied space in center of Building 3
Q1-IA-35	4	1st	Conference room on side table (center of Building 4)
Q1-IA-28	6	1st	Storage room on north side near former stairway
Q1-IA-36	7	1st	Far east room
Q1-IA-37	7/8	1st	West room next to stairs
Q1-IA-21	7/8	Basement	Hallway near Bldg 7/8 Sump 2
Q1-IA-23	7/8	Basement	Far east room - middle of room near the floor drain
Q1-IA-25	7/8	Basement	Main room near Bldg 7/8 Sump 1
Q1-IA-42	8	2nd	To be determined - occupied tenant space near elevator
Q1-IA-43	8	3rd	To be determined - occupied tenant space near elevator
Q1-IA-40	9	1st	West side open space near utility room door
Q1-IA-41	9	1st	East side storage room
Q1-IA-22	10	Basement	Center of main room
Q1-IA-44	10	1st	Suite 1001 - to be determined
Q1-IA-45	10	1st	Suite 1003 - to be determined
Q1-IA-39	11	1st	West side of main room

Crawl Space Air Sample Locations

Location ID	Bldg #	Floor	Sample Location Description
Q1-CS-01	6	Crawl Space	Northwest side
Q1-CS-04	4	Crawl Space	South side
Q1-CS-05	3	Crawl Space	Hole in lobby tile floor, center of Bldg 3
Q1-CS-07	2	Crawl Space	South side

Outdoor Air Sample Locations

Location ID	Bldg #	Floor	Sample Location Description
Q1-OA-03	10	Fence	115 River Road south parking lot chained to fence
Q1-OA-09	1	Fence	South of 115 RR Bldg next to Hudson River
Q1-OA-10	12	Fence	Northwest corner of Building 12 at Quanta Site fence

TABLE 1B

Proposed Sample Locations - Winter 2013/2014 Vapor Intrusion Monitoring Event

163 Old River Road Building

Quanta Site, Edgewater, New Jersey

Indoor Air Sample Locations

Location ID	Sample Location Description
Q2-IA-01	Kitchen - counter top
Q2-IA-02	1st floor dining room - on table near wall
Q2-IA-03	2nd floor dining room - on table in SW room

Subslab Sample Locations

Location ID	Sample Location Description
Q2-VI-01	Storage room next to stairs
Q2-VI-02	Kitchen - north side next to water service closet

Outdoor Air Sample Locations

Location ID	Sample Location Description
Q2-OA-01	South side of 163 Old River Road building - chained to fence
Q2-OA-02	Northwest of parking lot - chained to fence

TABLE 1C

Proposed Sample Locations - Winter 2013/2014 Vapor Intrusion Monitoring Event

103 River Road Building

Quanta Site, Edgewater, New Jersey

Indoor Air Sample Locations

Location ID	Sample Location Description
Q3-IA-01	Medical office storage room
Q3-IA-02	Dentist office hallway by exit door
Q3-IA-03	Medical office reception area

Subslab Sample Locations

Location ID	Sample Location Description
Q3-VI-01	Medical office storage room
Q3-VI-02	South stairwell
Q3-VI-03	Medical office utility room

Outdoor Air Sample Locations

Location ID	Sample Location Description
Q3-OA-01	North side of 103 River Road building
Q3-OA-02	Southwest corner of the 103 RR Building

TABLE 2

NJDEP RALs

115 River Road, 163 Old River Road, and 103 River Road Buildings

Quanta Site, Edgewater, New Jersey

Cas #	Parameter Name	NJDEP Nonresidential RAL ($\mu g/m^3$)
71-43-2	Benzene	200
100-41-4	Ethylbenzene	500
91-20-3	Naphthalene	26
127-18-4	Tetrachloroethene[1]	360
79-01-6	Trichloroethene[2]	18
95-63-6	1,2,4-Trimethylbenzene	Not Available
108-67-8	1,3,5-Trimethylbenzene	Not Available
108-38-3	o-Xylene[3]	Not Available
NA	m&p-Xylene[3]	Not Available
1330-20-7	Xylenes (total) - sum of isomers	880

Notes:

NJDEP RALs are from Table 2 of the NJDEP (March 2013) Vapor Intrusion Screening Level Tables; however if the RALs are updated, then the most current RALs will be used. Also, if NJDEP releases Health Department Notification Levels (HDNLs) in the future, then they will also be utilized for indoor air data evaluation.

NC = Non-carcinogenic

C = Carcinogenic

NJDEP = New Jersey Department of Environmental Protection

RAL = Rapid Action Level

[1] = Only the 103 River Road samples will be analyzed for tetrachloroethene.

[2] = Only the 163 Old River Road and 103 River Road samples will be analyzed for trichloroethene.

[3] = o-Xylene and m&p-xylene will be added together and compared to the screening level for total xylenes.

TABLE 3A

EPA Risk-Based Indoor and Crawl Space Air Screening Levels

115 River Road, 163 Old River Road, and 103 River Road Buildings

Quanta Site, Edgewater, New Jersey

		EPA Industrial IASL		
Cas #	**Parameter Name**	**10^{-6} Target Risk (µg/m³)**	**10^{-4} Target Risk (µg/m³)**	**HQ=1 Target Risk (µg/m³)**
71-43-2	Benzene	1.6	160	130
100-41-4	Ethylbenzene	4.9	490	4,400
91-20-3	Naphthalene	0.36	36	13
127-18-4	Tetrachloroethene[1]	47	4,700	180
79-01-6	Trichloroethene[2]	3.0	300	8.8
95-63-6	1,2,4-Trimethylbenzene	NA	NA	31
108-67-8	1,3,5-Trimethylbenzene[3]	NA	NA	31
108-38-3	o-Xylene	NA	NA	440
NA	m&p-Xylene[4]	NA	NA	440
1330-20-7	Xylenes (total) - sum of isomers	NA	NA	440

Notes:

The EPA IASLs are based on the latest EPA Regional Screening Levels (May 2013) for industrial air; however if the RSLs are updated, then the most current RSLs will be used to develop IASLs.

NA = Not applicable

IASL = Indoor Air Screening Level

[1] = Only the 103 River Road samples will be analyzed for tetrachloroethene.

[2] = Only the 163 Old River Road and 103 River Road samples will be analyzed for trichloroethene.

[3] = An RSL is not available for 1,3,5-trimethylbenzene; the RSL for 1,2,4-trimethylbenzene will be considered an evaluation surrogate for 1,3,5-trimethylbenzene

[4] = m&p-Xylene will be added to o-xylene and compared to the screening levels for total xylenes.

TABLE 3B

NJDEP Indoor and Crawl Space Air Screening Levels

115 River Road, 163 Old River Road, and 103 River Road Buildings

Quanta Site, Edgewater, New Jersey

Cas #	Parameter Name	NJDEP Nonresidential IASL (µg/m³)
71-43-2	Benzene	2
100-41-4	Ethylbenzene	5
91-20-3	Naphthalene	3
127-18-4	Tetrachloroethene[1]	47
79-01-6	Trichloroethene[2]	3
95-63-6	1,2,4-Trimethylbenzene	Not Available
108-67-8	1,3,5-Trimethylbenzene	Not Available
108-38-3	o-Xylene[3]	Not Available
NA	m&p-Xylene[3]	Not Available
1330-20-7	Xylenes (total) - sum of isomers	440

Notes:

NJDEP Nonresidential IASLs are from the NJDEP Vapor Intrusion Screening Level Tables (March 2013)

IASL = Indoor Air Screening Level

NJDEP = New Jersey Department of Environmental Protection

[1] = Only the 103 River Road samples will be analyzed for tetrachloroethene.

[2] = Only the 163 Old River Road and 103 River Road samples will be analyzed for trichloroethene.

[3] = o-Xylene and m&p-xylene will be added together and compared to the screening level for total xylenes.

TABLE 4A

EPA Risk-Based Subslab Soil Gas Screening Levels

163 Old River Road and 103 River Road Buildings

Quanta Site, Edgewater, New Jersey

		Industrial SGSLs		
Cas #	**Parameter Name**	**10^{-6} Target Risk (µg/m³)**	**10^{-4} Target Risk (µg/m³)**	**HQ=1 Target Risk (µg/m³)**
71-43-2	Benzene	16	1,600	1,300
100-41-4	Ethylbenzene	49	4,900	44,000
91-20-3	Naphthalene	3.6	360	130
127-18-4	Tetrachloroethene[1]	470	47,000	1,800
79-01-6	Trichloroethene	30	3000	88
95-63-6	1,2,4-Trimethylbenzene	NA	NA	310
108-67-8	1,3,5-Trimethylbenzene[2]	NA	NA	310
108-38-3	o-Xylene	NA	NA	4,400
NA	m&p-Xylene[3]	Not Available		
1330-20-7	Xylenes (total) - sum of isomers	NA	NA	4,400

Notes:

The EPA SGSLs are based on the latest EPA Regional Screening Levels (May 2013) for industrial air; however if the RSLs are updated, then the most current RSLs will be used to develop SGSLs.

The SGSLs were derived from the EPA RSLs by applying the EPA Vapor Intrusion Guidance (November 2002) default attenuation factor of 0.1.

NA = Not applicable

SGSL = Soil Gas Screening Level

[1] = Only the 103 River Road samples will be analyzed for tetrachloroethene.

[2] = An RSL is not available for 1,3,5-trimethylbenzene; the RSL for 1,2,4-trimethylbenzene will be considered an evaluation surrogate for 1,3,5-trimethylbenzene

[3] = m&p-Xylene will be added to o-xylene and compared to the screening levels for total xylenes.

TABLE 4B

NJDEP Subslab Soil Gas Screening Levels

163 Old River Road and 103 River Road Buildings

Quanta Site, Edgewater, New Jersey

Cas #	Parameter Name	NJDEP Nonresidential SGSL ($\mu g/m^3$)
71-43-2	Benzene	79
100-41-4	Ethylbenzene	250
91-20-3	Naphthalene	26
127-18-4	Tetrachloroethene[1]	2,400
79-01-6	Trichloroethene	150
95-63-6	1,2,4-Trimethylbenzene	Not Available
108-67-8	1,3,5-Trimethylbenzene	Not Available
108-38-3	m-Xylene[2]	Not Available
NA	o&p-Xylene[2]	Not Available
1330-20-7	Xylenes (total) - sum of isomers	22,000

Notes:

NJDEP Nonresidential SGSLs are from Table 1 of the NJDEP Vapor Intrusion Screening Level Tables (March 2013)

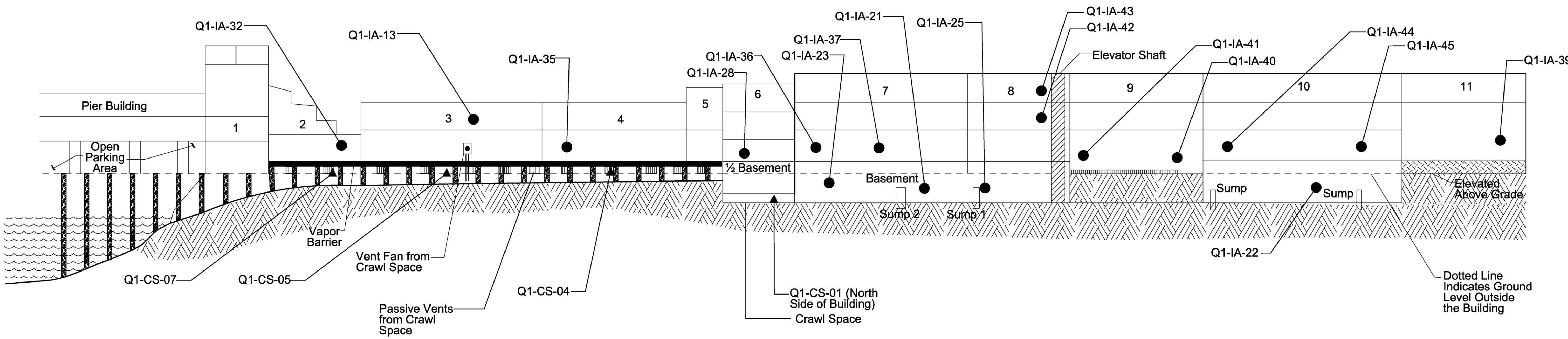
SGSL = Soil Gas Screening Level

NJDEP = New Jersey Department of Environmental Protection

[1] = Only the 103 River Road samples will be analyzed for tetrachloroethene.

[2] = o-Xylene and m&p-xylene will be added together and compared to the screening level for total xylenes.

Figures



LEGEND

● Q1-IA-07 Indoor Air Sample Locations

▲ Q1-CS-07 Crawl Space Air Sample Locations

NOTE

Building lengths approximately to scale. Other building features not to scale.

0 25 50 75
Scale In Feet

CH2MHILL

115 River Road Building
Winter 2013 - 2014
Vapor Intrusion Monitoring Event
Proposed Sampling Locations

Quanta Resources Superfund Site
115 River Road Building
Edgewater, New Jersey

December 2013	Figure 1

115 rr building_November_2013.dgn

* Sample Locations are approximate.

LEGEND

- Quanta Property Boundary
- Q1-IA-07 — Indoor Air Sample Locations
- Q1-OA-03 — Outdoor Air Sample Locations
- Q1-CS-01 — Crawl Space Air Sample Locations

NOTES

1. Source of Aerial Photograph: NJ Office of Information Technology (NJOIT), Office of Geographic Information Systems (OGIS), 2007-2008, Scale: 1:2400 (1"=200'), March - May 2007.
2. Approximate property boundaries taken from most recent Bergen County Tax Maps.
3. For purposes of this figure property lines are not extended into the Hudson River.

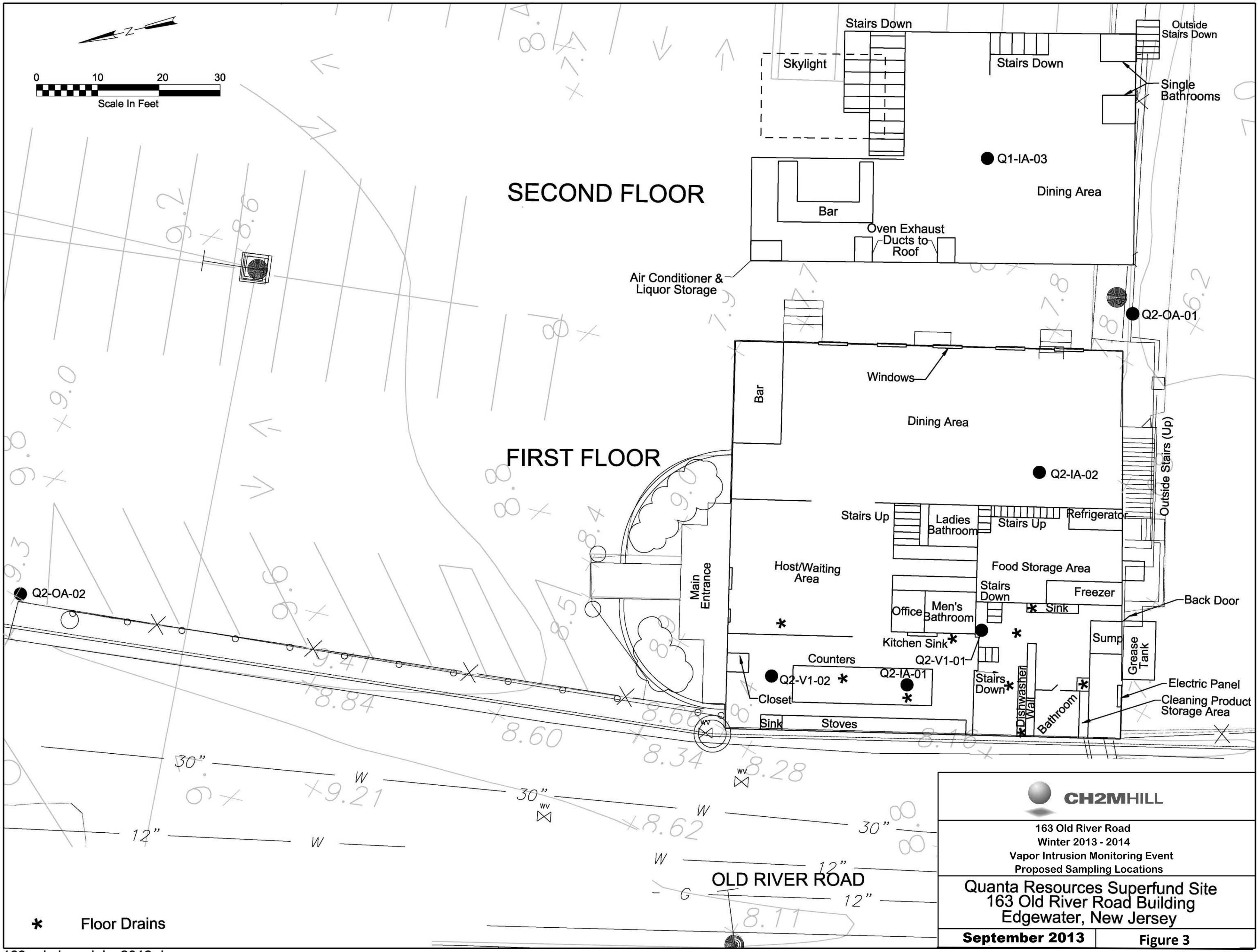
CH2MHILL

115 River Road Building
Winter 2013 - 2014
Vapor Intrusion Monitoring Event
Proposed Sampling Locations

Quanta Resources Superfund Site
115 River Road Building
Edgewater, New Jersey

November 2013	Figure 2

Proposed OA_IA_Samples_November_2013.dgn



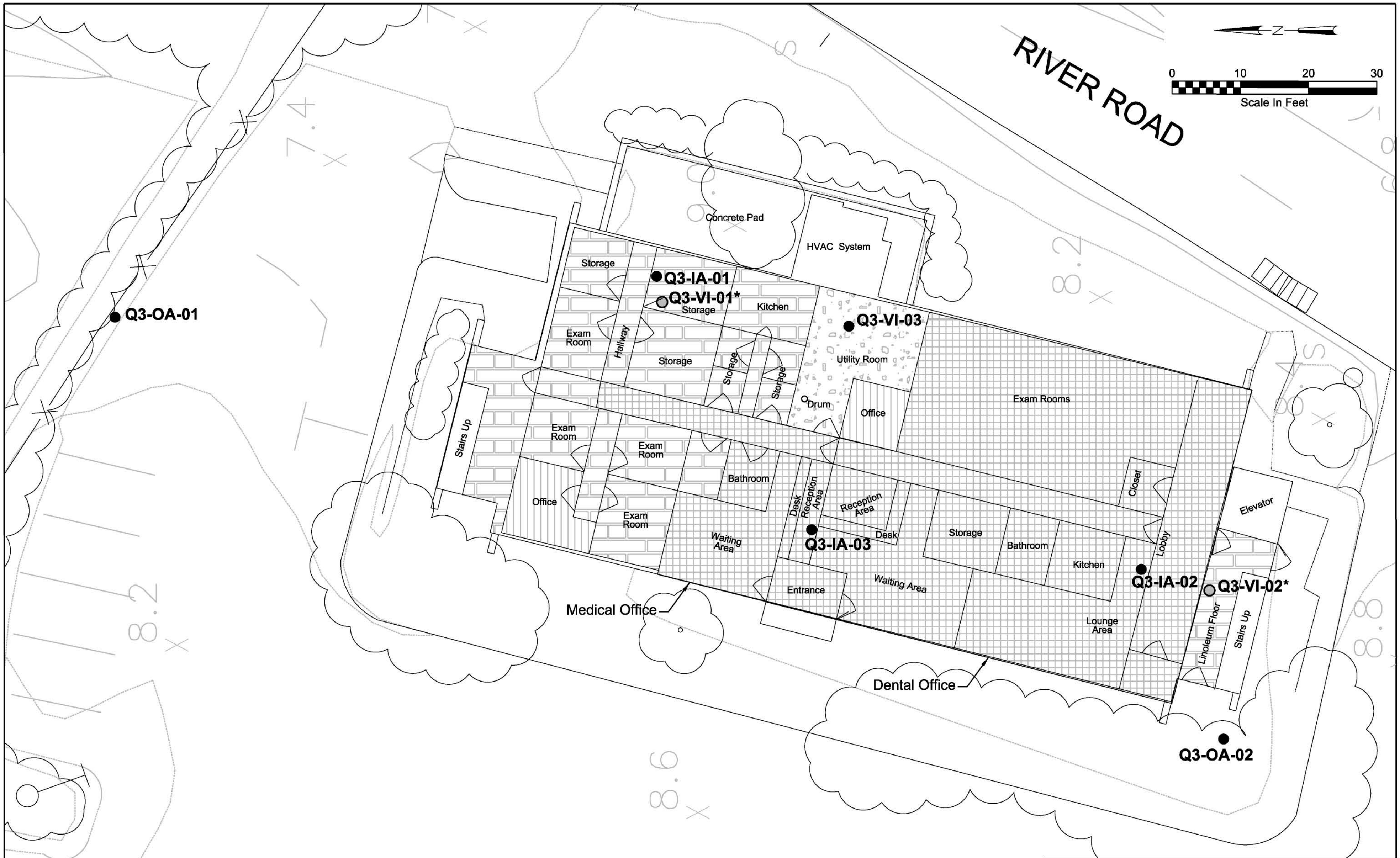
163 Old River Road
Winter 2013 - 2014
Vapor Intrusion Monitoring Event
Proposed Sampling Locations

Quanta Resources Superfund Site
163 Old River Road Building
Edgewater, New Jersey

September 2013

Figure 3

163 rr ia locs_july_2012.dgn



* These two subslab soil gas probes were found to be rusted shut in March 2013 due to flooding from Hurricane Sandy. They will be reinstalled and sampled during the winter 2013-2014 monitoring event.

Sample Locations are approximate.

Medical office room locations are close to scale.
Dental office room locations are approximate.

Carpet

Ceramic Tile

Linoleum Tile

Bare Concrete

CH2MHILL

103 River Road Building
Winter 2013 - 2014
Vapor Intrusion Monitoring Event
Proposed Sampling Locations

Quanta Resources Site
103 Old River Road Building
Edgewater, New Jersey

September 2013 | Figure 4

103_RR_September_2013.dgn

Attachment A
Responses to NJDEP's July 2013 E-mail Comments

ATTACHMENT A

Response to Comments

This attachment presents responses to comments from the New Jersey Department of Environmental Protection (NJDEP) on the letter report (CH2M HILL, 2013) about the results of the 2013 vapor intrusion (VI) monitoring events at the 115 River Road, 163 Old River Road, and 103 River Road buildings at the Quanta Resources Corporation Superfund Site (site), Operable Unit 1 (OU1). These comments were forwarded to Honeywell International Inc. (Honeywell) via email on July 23, 2013, following the meeting with Honeywell, CH2M HILL, NJDEP, and the U.S. Environmental Protection Agency (USEPA). At that meeting, the agencies agreed with Honeywell's proposal to submit responses to NJDEP's comments as an attachment to the work plan for the winter 2013/2014 VI monitoring event, and revisions to the 2013 monitoring report would not be required.

General Comments

Comment 1:

The document states on page 2 that when there is disparity between the USEPA and the NJDEP, the USEPA guidance and/or USEPA Region II standard practices will take precedence since Region II is the lead regulatory agency for the site. The NJDEP may not concur with proposed plans, reports or remedial actions for the site should these items fail to comply with the Department's regulatory requirements or departmental guidance applicable to the site.

Response to Comment (RTC) 1:

This detail was added to clarify if discrepancies were to occur, USEPA practices would be followed; however, it should be noted that, historically, the sample collection and analytical procedures used for the VI monitoring at the site are consistent with NJDEP (2013) VI guidance, with a single variation made at the specific direction of the USEPA. Richard Ho/USEPA directed Honeywell in 2011 to collect subslab soil gas samples over a 24-hour period. This direction was stated as being in accordance with USEPA Region II standard practices. This differs from the NJDEP (2013) Vapor Intrusion Technical (VIT) Guidance, which recommends collecting grab subslab samples after indoor air sampling is completed to avoid potentially impacting indoor air samples with soil gas. Other than this one variation at USEPA's direction, all sampling, analytical, and data submittal are being performed in accordance with the NJDEP (2013) VIT Guidance.

VI Results Comments for 115 River Road

Comment 2:

The indoor air analytical results for Buildings 6, 7, 8, and 9 exceed the March 2013 NJDEP nonresidential IASL for benzene (2 $\mu g/m^3$) and/or naphthalene (3 $\mu g/m^3$) with levels ranging up to 5.9 $\mu g/m^3$ and 9.3 $\mu g/m^3$, respectively. An elevated ethyl benzene level of 7.2 $\mu g/m^3$, above the NJDEP nonresidential IASL of 5 $\mu g/m^3$, was also detected in sample IA-41 in Building 9. Consistent with previous results, the higher levels are present in the Building 7/8 basement (see IA-21 and IA-25), with elevated levels also noted in sample IA-41 in Building 9 located closest to the basement of Building 7/8 and the elevator shaft. The indoor air sampling survey indicates that portions of Building 9 are above the Building 7/8 basement and sample IA-41 was located in the area with an odor similar to that found in the Building 7/8 basement. The survey states Building 9 is currently occupied by the Osteo Relief Institute.

The elevated contaminant levels detected in the buildings must be addressed consistent with section 1.15(e) of the Technical Requirements for Site Remediation (N.J.A.C. 7:26E), which requires vapor mitigation and a monitoring plan. DEP has concerns as to the effectiveness of the interim ventilation system in the Building 7/8 basement since the system was noted to be turned off in the report. The proximity to the elevator shaft, detection of odors and the presence of the Building 7/8 basement under Building 9 also raises questions as to potential impacts to the indoor air quality of Building 9. As noted in previous DEP comments, VI sampling should target areas of preferential pathways, such as elevator pits, as outlined in section 3.5 of the NJDEP Vapor Intrusion Technical (VIT) Guidance, March 2013.

A meeting between the agencies and PRPs is planned. During the meeting, it should be clarified when the buildings will be demolished and what additional measures (e.g., increased ventilation, sealing of entry points/cracks) can be instituted to ensure that the VI pathway is adequately addressed while the buildings are still present. Portions of the buildings that are currently occupied are of greatest concern in terms of the VI pathway and ensuring the public's health and safety. These areas should be targeted for additional mitigation measures and VI sampling.

RTC 2:

A vapor mitigation system was installed in the Building 7/8 basement in 2008, and consists of fans installed in the basement to increase the outdoor air exchange rate and ductwork added to vent airflow to the roof. Floor drains and sumps were covered or sealed to reduce vapor entry points in the basement. Historically, operation of this system was performed by the building owner, with oversight and technical support as needed provided by Honeywell. The after-effects of Hurricane Sandy temporarily reduced the effectiveness of the mitigation system, and the recent transaction of the property slowed the response to this issue. As such, in July 2013, Honeywell met with the new building owner about the need for operation of the system and recommended housekeeping of the basement. The owner was informed that a monthly inspection will be conducted of the Building 7/8 basement ventilation system as part of the indoor air monitoring program. These inspections will allow for necessary corrections to be made throughout the entire year.

The historical VI sampling results, along with the forthcoming 2013/2014 indoor air monitoring results, will be reviewed to determine the effectiveness of the mitigation system in reducing concentrations in the Building 7/8 basement. If needed, additional measures will be identified and implemented, as appropriate, to continue to reduce indoor air concentrations in the occupied tenant spaces prior to building demolition.

Honeywell also notified the owner about the indoor air results in Building 9, and informed the owner of the need to identify and seal potential vapor transport pathways in Building 9. During the 2013 sampling event, sealing foam was observed around the utility entries into the storage room on the west side of the Building 9 first floor. Honeywell recommended to the owner that that supplementary sealing around utility conduits and any other potential air pathways in the basement be performed to prevent basement air from intruding into this storage room. As described in the Work Plan for the 2013/2014 sampling event, this storage room will be resampled to determine if added sealing or mitigation measures are needed to reduce indoor air concentrations.

The Work Plan for the winter 2013/2014 sampling event (Author, year) includes proposed sampling locations to address NJDEP's concerns regarding the elevator shaft as a conduit. Indoor air samples will be collected from each of the occupied first floor tenant spaces at 115 River Road during the winter 2013/2014 VI monitoring event. Additional sampling will be performed at Building 8 to determine if the elevator shaft is acting as a preferential vapor pathway. Indoor air samples will be collected from the second and third floor occupied tenant spaces closest to the elevator. This sampling will be performed while the Basement 7/8 mitigation system is functioning, as described previously.

Currently, Honeywell anticipates that the 115 River Road building will be demolished in 2014/2015 to allow access to areas where in situ stabilization will be performed. In the meantime, Honeywell has requested to be informed by the building owner of changes in building occupancy so that monitoring and mitigation measures can be adjusted accordingly. The proposed sampling locations were selected based on the current building occupancy as of September 2013, and will be modified as needed. Any updates to the sampling scheme will be communicated to USEPA.

VI Results Comments for 163 Old River Road

Comment 3:

The targeted indoor air and sub-slab soil gas analytical results are below the March 2013 NJDEP nonresidential screening levels. Figure 1 in Appendix H indicates elevated ground water trichloroethene (TCE) levels (up to 86 µg/L), above the Department's ground water screening level (GWSL) of 2 µg/L, have been detected in the area of the 163 Old River Road building. Future monitoring to be conducted at the site should be consistent with the Department's VIT guidance

RTC 3:

TCE is not a site related COC but will be included in the analyte list for the winter 2013/2014 VI monitoring event at the 163 Old River Road building. TCE was not detected in indoor air and subslab soil gas samples from the initial VI sampling event in March 2008 when the full TO-15 list was reported. Therefore, the subsequent USEPA-approved VI monitoring work plans for the 163 Old River Road building did not include TCE in the analyte list.

VI Results Comments for 103 River Road

Comment 4:

The targeted indoor air and sub-slab soil gas analytical results are below the March 2013 NJDEP nonresidential screening levels. Two of the three sub-slab sampling points were noted to be rusted due to hurricane Sandy and were unable to be sampled during the current sampling round. Replacement sampling points should be installed for future sampling rounds in accordance with DEP VIT Guidance. Appendix H indicates elevated TCE levels in ground water (up to 89.7 µg/L) have been detected near the 103 River Road building. Future monitoring to be conducted at the site should be consistent with the Department's VIT guidance.

RTC 4:

The two subslab sampling probes that were rusted shut in March 2013 will be replaced and sampled, and TCE will be included in the analyte list during the winter 2013/2014 VI monitoring event at the 103 River Road building. TCE was not detected in subslab soil gas samples from the initial VI sampling event in March 2009 when the full TO-15 list was reported. Therefore, the subsequent USEPA-approved VI monitoring work plans for the 103 River Road building did not include TCE in the analyte list.

References

CH2M HILL. 2013. *Quanta Resources Corporation Superfund Site, Operable Unit 1 (OU1) Vapor Intrusion—Results of the 2013 Monitoring Events at 115 River Road, 163 Old River Road, and 103 River Road.* May 21.

New Jersey Department of Environmental Protection (NJDEP). 2013. *Vapor Intrusion Technical Guidance* and the associated NJDEP Vapor Intrusion Screening Level Tables. March.

Attachment B
Standard Operating Procedures

Cox-Colvin & Associates, Inc. Environmental Services

Standard Operating Procedure
Installation and Extraction of the Vapor Pin™

May 20, 2011

Scope:

This standard operating procedure describes the installation and extraction of the Vapor Pin™[1] for use in sub-slab soil-gas sampling.

Purpose:

The purpose of this procedure is to assure good quality control in field operations and uniformity between field personnel in the use of the Vapor Pin™ for the collection of sub-slab soil-gas samples.

Equipment Needed:

- Assembled Vapor Pin™ [Vapor Pin™ and silicone sleeve (Figure 1)];
- Hammer drill;
- 5/8-inch diameter hammer bit (Hilti™ TE-YX 5/8" x 22" #00206514 or equivalent);
- 1½-inch diameter hammer bit (Hilti™ TE-YX 1½" x 23" #00293032 or equivalent) for flush mount applications;
- ¾-inch diameter bottle brush;
- Wet/dry vacuum with HEPA filter (optional);
- Vapor Pin™ installation/extraction tool;
- Dead blow hammer;
- Vapor Pin™ flush mount cover, as necessary;
- Vapor Pin™ protective cap; and
- VOC-free hole patching material (hydraulic cement) and putty knife or trowel.

[1] Cox-Colvin & Associates, Inc., designed and developed the Vapor Pin™; a patent is pending.

Figure 1. Assembled Vapor Pin™.

Installation Procedure:

1) Check for buried obstacles (pipes, electrical lines, etc.) prior to proceeding.

2) Set up wet/dry vacuum to collect drill cuttings.

3) If a flush mount installation is required, drill a 1½-inch diameter hole at least 1¾-inches into the slab.

4) Drill a 5/8-inch diameter hole through the slab and approximately 1-inch into the underlying soil to form a void.

5) Remove the drill bit, brush the hole with the bottle brush, and remove the loose cuttings with the vacuum.

6) Place the lower end of Vapor Pin™ assembly into the drilled hole. Place the small hole located in the handle of the extraction/installation tool over the Vapor Pin™ to protect the barb fitting and cap, and tap the Vapor Pin™ into place using a

dead blow hammer (Figure 2). Make sure the extraction/installation tool is aligned parallel to the Vapor Pin™ to avoid damaging the barb fitting.

Figure 2. Installing the Vapor Pin™.

For flush mount installations, unscrew the threaded coupling from the installation/extraction handle and use the hole in the end of the tool to assist with the installation (Figure 3).

Figure 3. Flush-mount installation.

During installation, the silicone sleeve will form a slight bulge between the slab and the Vapor Pin™ shoulder. Place the protective cap on Vapor Pin™ to prevent vapor loss prior to sampling (Figure 4).

Figure 4. Installed Vapor Pin™.

7) For flush mount installations, cover the Vapor Pin™ with a flush mount cover.

8) Allow 20 minutes or more (consult applicable guidance for your situation) for the sub-slab soil-gas conditions to equilibrate prior to sampling.

9) Remove protective cap and connect sample tubing to the barb fitting of the Vapor Pin™ (Figure 5).

Figure 5. Vapor Pin™ sample connection.

10) Conduct leak tests [(e.g., real-time monitoring of oxygen levels on extracted sub-slab soil gas, or placement of a water

dam around the Vapor Pin™) Figure 6]. Consult your local guidance for possible tests.

Figure 6. Water dam used for leak detection.

11) Collect sub-slab soil gas sample. When finished sampling, replace the protective cap and flush mount cover until the next sampling event. If the sampling is complete, extract the Vapor Pin™.

Extraction Procedure:

1) Remove the protective cap, and thread the installation/extraction tool onto the barrel of the Vapor Pin™ (Figure 7). Continue

Figure 7. Removing the Vapor Pin™.

turning the tool to assist in extraction, then pull the Vapor Pin™ from the hole (Figure 8).

Figure 8. Extracted Vapor Pin™.

2) Fill the void with hydraulic cement and smooth with the trowel or putty knife.

3) Prior to reuse, remove the silicone sleeve and discard. Decontaminate the Vapor Pin™ in a hot water and Alconox® wash, then heat in an oven to a temperature of 130° C.

The Vapor Pin™ to designed be used repeatedly; however, replacement parts and supplies will be required periodically. These parts are available on-line at www.CoxColvin.com.

Replacement Parts:

Vapor Pin™ Kit Case - VPC001
Vapor Pins™ - VPIN0522
Silicone Sleeves - VPTS077
Installation/Extraction Tool - VPIE023
Protective Caps - VPPC010
Flush Mount Covers - VPFM050
Water Dam - VPWD004
Brush - VPB026

Standard Operating Procedure for Conducting Building Surveys for Vapor Intrusion Evaluations

Quality Management

SOP NO. : BLDGSURVEY-1
ORIGINAL VERSION 07/08/2008 BY MEB
REVISION 1 – 02/08/2009 BY JNS
REVISION 2 - 01/17/2011 BY KAS
REVISION 3 - 12/16/2011 BY JNS
PAGES: 7

File Name: SOP_VI_Building_Survey_080711.Doc

Location: Vapor Intrusion SharePoint Site/Best Practices and OPs/Standard Operating Procedures (SOPs)

Primary Contact(s): Jennifer Simms/PHL and Ben Thompson/CVO

QA/QC Review: Dave MacPhaul/GNV, Mike Bedan/DEN, Julie Keating/DEN, Loren Lund/DEN

Associated Internal Resources:

Excel survey forms for commercial/industrial buildings and residential buildings are available at Vapor Intrusion SharePoint Site/Best Practices and OPs/Standard Operating Procedures (SOPs).

Example project reports using this SOP can be found at VI Practitioners Site (Limited Access)/Project Document Library

For vapor intrusion evaluations in residential areas, a community outreach plan should be developed and the field team should be trained on how to interact with residents. Internal *Public Involvement Specialists* who can be consulted for this include Amy Brand/VBO and Ginny Farris/WDC.

*** THIS PAGE IS FOR INTERNAL USE ONLY.
DO NOT DISTRIBUTE TO EXTERNAL PARTIES ***

Standard Operating Procedure for Conducting Building Surveys for Vapor Intrusion Evaluations

1. Purpose and Objectives

This standard operating procedure (SOP) presents general guidelines for conducting building surveys for vapor intrusion evaluations. A building survey is performed as part of a vapor intrusion evaluation to obtain information for development of a conceptual site model (CSM) and to prepare for vapor intrusion sampling (select optimal sampling locations and determine if there are any potential indoor sources of volatile organic compounds [VOCs]).

A CSM for vapor intrusion pathway evaluation describes potential constituent sources, migration pathways, and potential human receptors under current and/or future land uses at the site. The important building characteristics for vapor intrusion pathway evaluation include the following:

- building use and occupancy
- condition of the building envelope
- presence of a basement or crawl space
- dimensions of the building and interior compartments
- condition of the slab and basement walls and presense of potential vapor intrusion pathways
- type and typical operational settings of the heating, ventilation, and air conditioning (HVAC) system
- the presence of potential indoor sources of VOCs.

This SOP can be used to perform building surveys in residential, commercial, or industrial buildings. At project sites with multiple buildings, a building survey should be performed for each building that is included in the vapor intrusion evaluation.

2. Project-Specific Considerations

2.1 Some states include building survey procedures and forms in their regulations or guidance documents. It is the responsibility of the project team to make sure this procedure meets all applicable regulatory standards and receives approval/concurrence from the leading regulatory agency for the project.

2.2 The building survey will likely be the first interaction with the occupants at the building and is an appropriate time to provide occupants with information on the vapor intrusion evaluation being performed and any sampling procedures that will be used. For vapor intrusion evaluations in residential areas, a community outreach plan should be developed and the field team should be trained on how to communicate with residents.

2.3 Varying levels of detail can be attained for building surveys. The project should develop data quality objectives (DQOs) to determine what specific information should be collected for their project.

2.4 Ideally, the building survey should be conducted at least one week before the actual indoor air or subslab soil gas sampling event. This advance timeframe allows the vapor intrusion investigator to identify and eliminate (to the extent practical) potential background sources of indoor air contamination. It also permits the investigator to confirm the sample locations with the occupants and regulatory agency(s) (if applicable) ahead of the scheduled sampling event.

3. Health and Safety

There are several health and safety topics to consider when performing building surveys:

3.1 Field teams should work in pairs at residential buildings or at industrial/commercial buildings where a relationship with the building occupant has not yet been established. A field team member should never enter a building alone for the first time. The mental stability of a building occupant should not be taken for granted. Building surveys at abandoned buildings should also be performed in pairs; if one team member is injured, the other will be able to seek help.

3.2 Walk slowly and with caution to avoid slips, trips, and falls.

3.3 Beware of animals and insects. This applies to abandoned buildings and residences.

3.4 Be careful of overhead hazards in basements. Do not attempt to enter crawl spaces.

4. Materials

4.1 Building Survey Form – to record survey information. Example forms are provided as attachments to this SOP. There is one for residential buildings, and one for industrial/commercial buildings.

4.2 Figure showing the footprint of the building (if available) – to mark up during the building survey with information about the building characteristics. It may also be helpful to ask the building contact for a copy of the fire evacuation map which will show the building footprint and interior walls.

4.3 Flashlight

4.4 Walking wheel or measuring tape – to measure building and room dimensions

4.5 Camera – to photograph the building (interior and exterior) if allowed by the building owner

4.6 Photoionization detector – to monitor total VOCs for health and safety at sites where high VOC concentrations may be expected (*OPTIONAL*)

5. Field Procedures

5.1. Building Survey

5.1.1. Gain access to the building. Schedule the site visit with the site contact. At a client- owned and -operated site, this step may just require a phone call to the client. At an off-site residence, this may require significant coordination, including obtaining an access agreement and providing vapor intrusion fact sheets to inform residents of the vapor intrusion pathway and the reason for the investigation.

5.1.2. Obtain occupant information. The building occupants are the potential receptors in the vapor intrusion CSM. Is the building use residential, commercial, or industrial? How many people typically occupy the building? Are there sensitive receptors (children, elderly, or immune-impaired) in the building? How much time do occupants spend in the building? What areas of the building do the occupants typically use (i.e., where do they spend the most time)?

5.1.3. Obtain building information. How old is the building? What was its original use? Have there been any additions or other significant modifications? Additions will likely have slabs that are separate from the original building. How many floors does the building have? Does the building have a basement? If so, how far does it extend below grade? Is the slab on grade? Is the slab elevated above the ground surface?

5.1.4. Survey the building envelope. The condition of the building envelope will determine the rate of outdoor to indoor air exchange. A high rate of outdoor air exchange can dilute soil gas that may be migrating into the building. Walk around the inside and outside of the building and record information on the building construction and condition. How many doors/windows/loading docks are there, what condition are they in, and are they typically left open or closed? What are the building construction materials?

5.1.5. Determine the indoor air volume and the location and volume of separate indoor air compartments within the building. If a building has a very large indoor air volume, soil gas migrating into the building may become quickly diluted. Measure the building dimensions (length, width, and height). Measure the dimensions of compartments or rooms within the building. How are rooms connected? Are interior doors typically kept open or shut? Are there separate compartments within the building (i.e., areas that are not connected to other areas such that the indoor air does not mix)? Indoor air sampling may be necessary in multiple rooms if the indoor air volume is not connected.

5.1.6. Observe the slab condition. The building slab is the barrier between subslab soil gas and the indoor air. How thick is the slab? What is the general condition of the slab? What is the floor covering in each room of the lowest floor (carpet, tile, etc.)?

5.1.7. Identify potential vapor intrusion pathways. The entry of organic vapors into a structure is caused by the infiltration of contaminants through the slab and walls that are in contact with the soil. Any openings, cracks, or penetrations in the slab or basement walls may be entryways for subslab soil gas. Are there any utilities that penetrate the slab or basement walls? Are they sealed properly? Are there cracks in the slab or basement walls? If so, note where these cracks are and their approximate size. Are there sumps? If so, note the dimensions of each and their typical operating conditions. Is the wall/floor juncture sealed well? Is there a french drain? Has the basement been waterproofed? Are there expansion joints in the slab? If so, note their condition.

5.1.8. Evaluate the HVAC system. The heating, air-conditioning, and ventilation (HVAC) system's operation can determine if the building is negatively or positively pressurized. If a building is negatively pressurized, then subslab gas will be pulled into the building; if the building is positively pressurized, subslab gas will not enter the building. Record the type/model of the systems and the typical operating conditions. Is there one air conditioning zone or multiple zones (look for multiple thermostats)? Does the HVAC system use radiant heat or forced air? If the HVAC system is forced air, where are the heating/cooling and return air vents? What is the HVAC system's fresh air intake? What is the heating fuel source (i.e., natural gas, oil, propane)? Are there ventilation fans? If so, note where and their typical operating conditions. Are there window air conditioning units?

5.1.9. Identify any existing vapor mitigation systems. Is there a radon mitigation system or other subslab depressurization system? Is there sealant on any cracks or crevices? Is there a sealant coat on the floor for vapor or water mitigation?

5.1.10. Sketch the building floor plan. Record all pertinent building characteristics for the vapor intrusion evaluation. Include building dimensions, locations of windows/doors/loading docks, outdoor surface cover (grass, asphalt, etc.), and locations of any potential indoor VOC sources.

5.1.11. Identify potential indoor contaminant sources within the building. Record the location of the potential sources and determine if they can be removed before indoor air sampling is performed. Potential indoor sources of VOCs may include cleaning products, paint, dry-cleaned clothes, gasoline, cosmetics, or cigarette smoke. Recent remodeling activities, including painting, installing new carpeting or flooring, and moving in new furniture should be identified, because they could be potential sources of VOCs. It may be necessary to include additional sheets to inventory all the potential VOC sources within the structure. Be sure to document any potential VOC sources that are removed from the structure so that it can be included in the data evaluation. When potential indoor VOC sources are identified and removed from a building, it may be necessary to ventilate the rooms affected in advance of the air sampling event. This ventilation should be completed at least 24 hours before the commencement of the indoor air sampling event. A hand-held field screening instrument can also be used to pinpoint potential indoor VOC sources.

5.1.12. Identify potential outdoor contaminant sources. These may include gas stations, major roadways, dry cleaners, repair shops, industries, or landfills.

5.2. Identify possible indoor air, outdoor air, crawl space air, and subslab soil gas sample locations that meet the project-specific DQOs. (OPTIONAL)

The selected sampling location(s) should be chosen in consultation with the property owner during the building survey.

Procedures for collecting indoor air, outdoor air, crawl space air, and subslab soil gas samples inside a building are described in the ***Standard Operating Procedure for Indoor, Outdoor, and Crawl Space Sampling for VOCs Using Canisters*** and the ***Standard Operating Procedure for Installing and Sampling Subslab Soil Gas Probes Using Canisters***.

5.2.1. Indoor Air Sample Locations

5.2.1.1. Typically, indoor air samples should be collected from each compartment or HVAC zone within a building.

5.2.1.2. Typically, indoor air samples should be collected on the lowest floor of the building at breathing zone height (approximately 3 to 5 feet) toward the center of the building away from windows.

5.2.1.3. Consideration should be given on a case-specific basis to those situations (such as a daycare facility) where a different sampling height may also be appropriate to evaluate a unique setting or population.

5.2.1.4. Indoor air samples should be located in the areas of the building that are occupied most frequently and by the most amount of people.

5.2.1.5. Indoor air samples can be collected from more than one floor within a structure to address varying risk exposures and as part of the process to distinguish contaminants related to vapor intrusion from background sources. Thus, the location and position of the sample container will vary depending on which floor the sampling event takes place.

5.2.1.6. The basement sample(s) are primarily designed to investigate "worst case" situations within a structure. Therefore, basement samples are positioned as close as possible to the source area (e.g., sumps or major cracks in the foundation).

5.2.2. Outdoor Air Sample Locations

5.2.2.1. Typically, outdoor air samples are collected upwind and/or downwind of the building or site being investigated.

5.2.2.2. Avoid biasing the sample results by placing the canister near potential outdoor VOC sources such as busy roads or gas stations.

5.2.2.3. Outdoor air samples are typically located at least 10 feet away from buildings. However, the outdoor air canister may be placed near the outdoor air intake for the HVAC system for the building.

5.2.2.4. Outdoor air sample canisters should be secured to an immovable structure to ensure security for sampling in public areas. A bicycle lock or piece of chain and padlock can be used. NOTE: Do not secure the canister to or close to a living tree, however, because the tree's evapotranspiration process may release VOCs from groundwater into the vicinity. It may be a good idea to attach a label to the canister explaining that it is an environmental sample and should not be tampered with. The label can also include contact information.

5.2.2.5. Typically, outdoor air samples should be collected at breathing zone height (approximately 3 to 5 feet).

5.2.3. Crawl Space Air Sample Locations

5.2.3.1. Crawl space air samples are typically collected in locations selected to achieve adequate spatial coverage of the building's crawl space. Sample location selection will be limited by accessibility.

5.2.3.2. Crawl space air sample inlets should be located several feet from the opening or access point to avoid dilution by outdoor air. In cases where the crawl space is most conveniently sampled by access through crawl space vents, a sampling probe (sample delivery line made of Teflon® or stainless steel) of sufficient length is attached to the inlet of the flow controller.

5.2.4. Subslab Soil Gas Sample Locations

5.2.4.1. Subslab soil gas sample locations should also be toward the center of the building and ideally in an area of exposed concrete away from any penetrations in the slab. Positions near the perimeter of the slab are subject to dilution and should be avoided. As a genereal rule, it is best to stay at least 5 to 10 feet away from any exterior wall.

5.2.4.2. Typically, subslab soil gas sample locations are biased towards areas of the building where the highest subsurface VOC concentrations are expected.

5.2.4.3. Typically, subslab soil gas sample locations should be spread out throughout the building to achieve adequate coverage of the entire building.

5.2.4.4. Make sure the proposed subslab soil gas sample density is in accordance with applicable regulatory guidance documents. Recommendations about how many subslab soil gas samples to collect vary, ranging from one subslab soil gas sample for every 330 square feet (or two to three samples for every average-sized home) to one subslab soil gas sample for an average residential dwelling of 1,500 square feet; however, a lesser denisty for very large building is usually acceptable.

5.2.4.5. To minimize potential damage to flooring, it may be necessary to select a location in a closet or utility room (where carpeting or tiles are less visible or not present at all).

6. Data Reduction and Evaluation

The information collected during the building survey can be used to develop a preliminary vapor intrusion CSM for the work plan, refine an existing CSM, select locations for indoor air and subslab samples, or to provide information to support the evaluation of the vapor intrusion pathway in a vapor intrusion evaluation or human health risk assessment.

7. Quality Control

Adequate time should be reserved for performing building surveys and detailed notes should be recorded at the time of the building survey.

8. Attachments

8.1. Residential Building Survey for Vapor Intrusion Evaluation Form

8.2. Industrial/Commercial Building Survey for Vapor Intrusion Evaluation Form

Standard Operating Procedure for Installing Subslab Probes and Collecting Subslab Soil Gas Samples Using Canisters

Quality Management

SOP NO. : SUBSLAB_SAMPLING_1

ORIGINAL VERSION 10/01/2005 BY BFT
REVISION 1 – 06/23/2008 BY BFT
REVISION 2 - 02/19/2009 BY BFT
REVISION 3 - 01/11/2011 BY KAS
REVISION 4 - 08/07/2011 BY JNS
REVISION 5 – 01/19/2012 BY JNS
REVISION 6 – 06/14/2012 BY JAL

PAGES: 18

File Name: SOP_Subslab_Soil_Gas_Installation_and_Sampling_6142012.Doc

Location: Vapor Intrusion SharePoint Site/Best Practices and OPs/Standard Operating Procedures (SOPs)

Primary Contact(s): Jennifer Simms/PHL, Ben Thompson/CVO, Kim Stokes/DFW, and John Lowe/SPK

QA/QC Review: Loren Lund/DEN, Shirley Steinmacher/SLC, and Kim Stokes/DFW

Associated Internal Resources:

Excel sampling logs are available at Vapor Intrusion SharePoint Site/Best Practices and OPs/Standard Operating Procedures (SOPs).

Example project reports using this SOP can be found at VI Practitioners Site (Limited Access)/Project Document Library

For vapor intrusion evaluations in residential areas, a community outreach plan should be developed and the field team should be trained on how to interact with residents. Internal *Public Involvement Specialists* who can be consulted for this include Amy Brand/VBO and Ginny Farris/WDC.

*** THIS PAGE IS FOR INTERNAL USE ONLY.
DO NOT DISTRIBUTE TO EXTERNAL PARTIES ***

Revision Log

REVISION 6 – 06/14/2012: 1) added specification for a calibrated digital vacuum gauge; 2) revised language for measuring and interpreting initial field vacuum measurements; 3) revised language for measuring and interpreting final field vacuum measurements; 4) added requirement for posting an informational sign on canisters being left unattended; 5) revised language and a revised table for estimating initial and final vacuums at different elevations.

** THIS PAGE IS FOR INTERNAL USE ONLY.
DO NOT DISTRIBUTE TO EXTERNAL PARTIES ***

Standard Operating Procedure for Installing Subslab Probes and Collecting Subslab Soil Gas Samples Using Canisters

1.0 Scope and Application

This standard operating procedure (SOP) describes the approach for installing subslab soil gas probes and collecting subslab soil gas samples using canisters (e.g., SUMMA canisters or equivalent). It includes instructions on probe installation, leak checking, soil gas sampling, and probe abandonment. This procedure should be used in conjunction with project data quality objectives. The project team is responsible for ensuring this procedure meets all applicable regulatory standards and receives approval/concurrence from the leading regulatory agency for the project. Vapor intrusion (VI) subject-matter experts (SMEs) should be consulted as needed to address technical, regulatory or field implementation issues associated with the use of this SOP. Only persons trained in the collection of subslab samples should attempt this procedure.

2.0 Project-Specific Considerations

2.1 A utility clearance must be performed prior to drilling through the slab, as with all intrusive site work. In addition, it is highly recommended that ground penetrating radar (GPR), specifically a concrete scanner (small, hand-held GPR unit designed for use inside buildings), be used to identify utilities, wire mesh, and/or rebar in the slab prior to drilling. The sampling team should look around the building to locate where utilities come into the building. Utility shut-off valves should be located in case an underground utility is encountered.

2.2 The Swagelok® parts (sampling union and nuts) may be re-used if they are decontaminated. Options for decontamination include: 1) purging with ultra pure air, 2) washing with alconox followed by hot water rinse, or 3) washing with methanol followed by hot water rinse. It is also advisable to heat the parts in an oven to a temperature of 130 degrees C (266 degrees F) after rinsing with water. The appropriate decontamination process should be selected during the work planning phase for each project. Typically subslab soil gas sampling does not generate investigation-derived waste (IDW) other than items that can be disposed of as solid waste; however, decontaminating with liquids will generate IDW. Compare the cost of buying new parts to the cost of managing and disposing of the IDW. The Teflon tubing cannot be resused or decontaminated.

2.3 There are three types of probe installation techniques. The type chosen depends on site access, probe seal integrity considerations, and the number of sampling events planned. It is critical that the sealing compound used is low in volatile organic compounds (VOCs). The following suggested sealing compounds below have been tested and approved for use. Consult a subject matter expert if another compound is preferred or available. See Table 1 for more specific details.

2.3.1 Temporary – Beeswax – Use if time is short, access is an issue, and a higher risk of leaks (requiring repeated resealing of the probe) is acceptable. It MUST be 100 percent pure, natural beeswax.

2.3.2 Semi-Permanent – Fix-It-All – Use if setting the probe and sampling in one day is preferred, access limitations are minimal, only one sampling event is intended, and minimal moisture is present.

2.3.3 Permanent – Portland cement – Use if there is unlimited access and multiple sampling events are desired.

TABLE 1
Probe Seal Types

Probe Type	Suggested Probe Seal	Benefits	Drawbacks
Temporary	Beeswax	Quick. Can Set probe and take sample in one visit Easy to remove	Wax is brittle when cool and is very susceptible to leakage.
Semi-permanent	Fix-It-All	Sets up fairly quickly (>30 min.), but may require 2 visits on the same day Solid seal Easy to remove	Not good for wet environments. Material breaks down
Permanent	Portland cement	Solid permanent seal Good for multiple sampling events	Takes at least 24 hours to set. Will require at least 2 visits on consecutive days Difficult to remove

3.0 Health and Safety

There are several health and safety topics to consider when installing and sampling subslab soil gas probes:

3.1 Field teams should work in pairs at residential buildings or at industrial/commercial buildings where a relationship with the building occupant has not yet been established. A field team member should never enter a building alone for the first time. The mental stability of a building occupant should not be taken for granted. Probe installation should also be performed in pairs.

3.2 The hammer drill is a large and powerful hand tool. When drilling, do not apply downward pressure, allow the drill to do the work. The drill bit is likely to become stuck if the operator is pushing down on the drill. Be prepared for the drill bit to catch and for the drill to stop suddenly; it can twist the operators wrist badly if unexpected.

3.3 Have a photoionization detector ready to screen the breathing zone during installation and sampling. Significant VOC concentrations may be present in subslab soil gas.

3.4 Beware of pinch points and use the correct hand tools to avoid hand injuries.

4.0 Canister Security

4.1 Field teams should assure that sampling canisters are not disturbed by building occupants.

4.2 If there is a community outreach program associated with the VI sampling event, then information should be made available to building occupants prior to the sampling event that informs occupants about the sampling activities and sampling equipment.

4.3 Each sampling canister should be clearly marked with a sign that includes contact information for a point of contact An example of a sign that can be attached to each sampling canister is provided in the attachment to this SOP. This sign can be edited with project-specific information, laminated and attached to each sampling canister using cable ties (do not attach the signs using adhesive tape).

5.0 Materials

5.1 Subslab Soil Gas Probe Installation

- Hammer drill and drill bits (7/8-inch and 1-inch OR 5/16-inch and 1/2-inch). NOTE: It is a good idea to have an extra-long drill bit available to drill through the occasional extra-thick slab.
- Vacuum cleaner (shop vac type or handheld, with HEPA filtration) for removing concrete dust generated while drilling through the slab for probe installation. Continuously vacuum the dust as it is generated during the drilling process.
- Subslab soil gas probe (for permanent or semi-permanent installations) See Figure 1 for an expanded view of the probe parts.
  - 1/4-inch outer diameter (OD) stainless steel tube for probe (Swagelok® part #SS-T4-S-035-20)
  - Swagelok® nut and ferrule set (part #SS-400-NFSET)
  - Probe union (1/4-inch male Swagelok® to 1/8-inch female NPT – part #SS-400-7-2).
  - Probe cap (Napa Auto Parts #3150X2 or Swagelok® part SS-2-HP)
- Metal tubing cutter for adjusting the length of the probe so that the probe does not extend below the slab
- Probe seal consisting of beeswax, Fix-it-All, or portland cement
- Wax melter (for beeswax only) – can be obtained from a beauty supply store (paraffin wax melter or body hair wax melter). Also need a clean metal measuring cup with handle for placing the wax into the melter; this way the wax can be melted in the cup and then easily poured into the probe hole. The beeswax CANNOT be melted with a direct flame because this generates VOCs and particulate pollutants.
- Large cotton swabs or paper towels and non-chlorinated (de-ionized or distilled) water for cleaning the concrete dust out of the hole
- Tongue depressor, putty knife, or similar tool for putting the probe seal material into the hole
- Teflon® pipe tape to wrap the end of the probe tubing so that the probe fits tightly into the hole to prevent the seal material from clogging the probe
- Tape measure to measure the thickness of the slab (measured off of a long screwdriver or drill bit)
- Optional (required by some regulatory agencies): glass “seed beads” (available at a craft store) to fill the void space created in the subslab during installation

- Optional: Sonicare® toothbrush with bristles removed. This can be useful in removing air bubbles from the cement mixture while installing the probe, thus making a more competent seal. Toothpicks or cotton swabs without cotton tip can also be used for this purpose.

FIGURE 1
Probe Parts

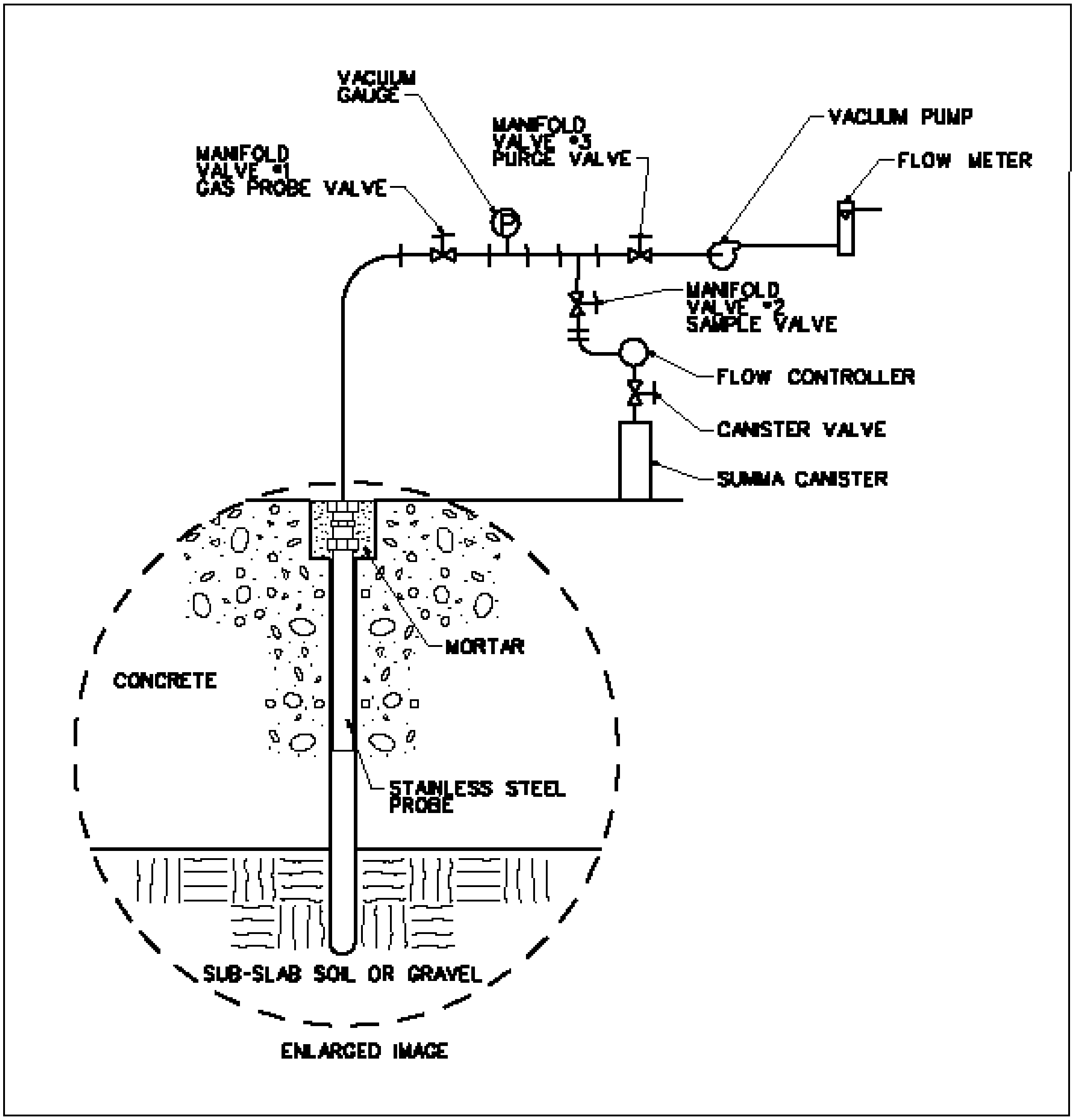
5.2 <u>Helium Leak Check</u>

- Helium canister containing high-grade helium (NOT balloon grade) and regulator for the canister (should be set to a flow rate of 200 milliliters per minute [mL/min] or less)
- Enclosure, which may be constructed from a small bowl or container
- Helium detector (e.g. Dielectric MGD®-2002), which can be rented from an equipment rental company.

5.3 <u>Subslab Soil Gas Sampling</u>

The subslab soil gas sampling set up is shown in Figure 2.

FIGURE 2
Subslab Soil Gas Sampling Diagram



- Sampling union: 1/4-inch male Swagelok® (or equivalent) to 1/4-inch male NPT (part # SS-400-1-2) (not necessary for beeswax method)
- Vacuum pump for purging with rotometer to control flow to 200 mL/min (should be a Cole Parmer # R-79200-00 grey diaphragm pump or equivalent)
- Sampling manifold consisting of Swagelok® gas-tight fittings with three valves and one vacuum gauge to attach the probe to the air pump and the sample canister. See

Figure 10. This manifold must be clean, free of oils, and flushed free of VOCs before use. This is accomplished by flushing three or four volumes of purge gas (ultra-high-purity [UHP] air or nitrogen) through the manifold and associated tubing.

- Swagelok® valve (only necessary for extended sampling periods [e.g., 8- or 24-hours] so that the sampling manifold can be disconnected without introducing indoor air into the probe) (part # SS-4P4T)
- Teflon® (or inert nylon) tubing, 1/4-inch outer diameter (OD)
- Tedlar® bag (1-L or 3-L) to collect the purged soil gas, so: (1) it is not discharged into the building, (2) the volume of purged soil gas can be measured, and (3) field screening with a PID or GEM2000 meter can be performed on the purged gas
- GEM2000® Landfill Gas Meter – this is optional if field measurements of CO_2, O_2, or CH_4 are necessary (aerobic biodegradation parameters typically measured for petroleum hydrocarbon sites)
- MiniRae® PID Meter – for health and safety to ensure breathing zone VOC concentrations remain below levels specified by the health and safety plan. It is also optional to collect field measurements of total VOCs from the probe or purged soil gas; may warn the lab if high concentrations are detected so they can dilute the sample before analysis.
- Canister, stainless steel, polished, certified-clean, and evacuated. These are typically cleaned, evacuated, and provided by the laboratory.
- Flow controller or critical orifice, certified-clean, and set at desired sampling rate. These are typically cleaned, set, and provided by the laboratory. Common sampling rates for subslab soil gas sampling are provided in Table 2.

TABLE 2
Common Sampling Rates for Subslab Soil Gas Sampling

Can Size	Length of Sampling Time	Sampling Flow Rate (mL/min)
6 Liter	1 hour	90
6 Liter	8 hours	11.25
6 Liter	24 hours	3.75
1 Liter	5 minutes	180
1 Liter	1 hour	15
850 ml	5 minutes	150
850 ml	1 hour	12

- Miscellaneous fittings (Swagelok® nut and ferrule, part #SS-400-NFSET) to connect tubing to sampling union and the canister
- Negative pressure (vacuum) gauge, oil-free and clean, to check canister vacuum. The vacuum gauges are typically provided by the laboratory. The laboratory may either provide one vacuum gauge to be used with all of the canisters, or a vacuum gauge for each canister to be left on during sample collection. Sometimes the canisters are fitted with built-in vacuum gauges that are not removable. Gauges sent by the laboratory are

for field use only, and are an approximate measure of the actual vacuum. Regularly calibrated – and less rugged – vacuum gauges are used at the laboratory to measure vacuum before shipment and again after sample receipt.

5.4 Probe Abandonment

- Probe removal fitting (or just use the sampling union)
- Crowbar
- Chissel and hammer
- Concrete patch (either pre-mixed cement patch or portland cement)

5.5 Miscellaneous

- Teflon® tape
- Modeling clay (VOC-free) for temporarily sealing probes that are leaking so the probe can be sampled and then patched with cement or Fix-It-All, or just abandoned for the beeswax method.
- Wrenches and screwdrivers (clean and free of contaminants) of various sizes as needed for connecting fittings and making adjustment to the flow controller. A 9/16-inch wrench fits the 1/4-inch Swagelok® fittings, which most canisters and flow controllers have.
- Extension cord
- Timer/watch
- Tools required to cut carpet and/or tools needed for removal of other floor coverings
- Shipping container suitable for protection of canister(s) during shipping. Typically, strong cardboard boxes are used for canister shipment. The canisters should be shipped to the laboratory in the same shipping container(s) in which they were received.

6.0 Subslab Soil Gas Probe Installation Procedure

6.1 Locate the sampling locations in accordance with the work plan. Note the location of the probe, locations of significant features (walls, cracks, sumps, drains, etc), and condition of the slab.

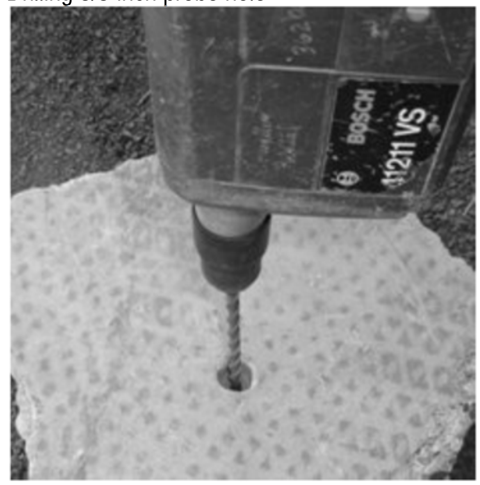
6.2 If needed, expose the concrete by cutting the carpet or other loose floor coverings (Note: carpet need not be removed, but rather an 'L' shape should be cut to expose the concrete for drilling and the leak-check enclosure).

6.3 Drill a 7/8-inch or 1-inch diameter hole to a depth of 1 and 3/4 inches (measured to the center of the hole) to allow room for installing the probe nut and probe union (See Figures 2 and 3). Remove the cuttings using the HEPA vacuum cleaner. Be careful to not compromise the integrity of the slab during drilling (e.g., cracking it), although make a note if this occurs. It

FIGURE 3
Drilling 1-inch mortar hole to a depth of 1 and 3/4-inch

is important that the slab and the probe hole remain airtight for sampling and that cracks are noted.

FIGURE 4
Drilling 3/8-inch probe hole



6.4 Drill a 5/16-inch or 1/2-inch diameter hole through the remainder of the slab and approximately 3 inches down into the subslab material (See Figures 2 and 4). Drilling into the subslab material creates a void that is free of obstructions that might plug the probe during sampling. Record the total depth of the slab and the depth drilled into the subslab material on the Subslab Soil Gas Sampling Log.

6.5 Clean out the drilled hole with the HEPA vacuum (equipped with a micro tip), cotton swabs, and paper towel. This removes any remaining dust, allowing the seal material to adhere to the hole wall better.

6.6 Some agencies may require that glass beads be poured into the subslab hole before installing the probe. If so, pour clean glass “seed beads” (available at a craft store) into the hole until enough beads have been added so that the top of the beads are even with the bottom of the slab. A thin piece of wire marked with the slab thickness and inserted into the hole can be used to determine this.

6.7 Install the subslab probe into the hole. First, trim the probe to the appropriate length so that, when inserted into the hole, it will not extend below the slab. Then wrap the end of the probe tubing with Teflon tape so that the probe fits tightly into the hole to prevent the seal material from clogging the probe. For permanent or semi-permanent probes, the probe is constructed of stainless steel tubing and Swagelok® parts. Temporary probes consist of 1/4-inch OD Teflon® tubing.

6.7.1 Temporary Seal (beeswax)

6.7.1.1 Melt the beeswax in the wax melter and pour the melted wax into the hole around the tubing. Be sure to get wax on all sides of the smaller diameter hole by moving the sample tube away from the walls. Continue to add wax until the hole is completely full.

6.7.1.2 Let the wax cool for 10 minutes.

6.7.1.3 Be sure to never leave the probe hole open to the atmosphere for extended periods to minimize the effects of surface infiltration.

6.7.1.4 <u>Be careful to never put too much force on the sampling tube.</u> The wax is only a temporary seal, and its sealing integrity can be compromised easily.

6.7.2 Semi-Permanent (Fix-It-All) or Permanent (portland cement) Seal

6.7.2.1 Wet the walls of the hole using a cotton swab or moistened paper towel. This helps the mortar bond to the drilled concrete. Prepare the mortar in accordance with manufacturer’s directions to a stiff consistency. Make sure that the consistency is such that the mixture will not run down the sides of the hole and potentially clog the probe or hole but is still easy enough to work with (so it can be easily scooped into the hole). Only mix an amount that can be used in 15 minutes. Place sample probe part-way into the hole, as shown in Figure 5. Using the tongue depressor or similar tool, apply mortar around the base of the sampling probe and sampling union such that it will be sealed once it is in place.

6.7.2.2 Fill the hole with mortar and press the probe further into the hole until its top is flush with the floor. In doing so, slightly wiggle the probe to create good 'wetting' contact between the probe and the mortar as well as the mortar and the drilled concrete. It may be helpful to work the concrete with a Sonicare® toothbrush (with the bristles removed) or a toothpick or similar object during this step to remove the air bubbles from the mortar and make a more effective seal. Scrape off excess and make sure there is clear access to the probe. See Figure 6.

6.7.2.3 For Fix-It-All, let dry for 30 minutes. For cement, let cure for 24 hours.

6.7.2.4 Be sure to never leave the probe hole open to the atmosphere for extended periods to minimize the effects of surface infiltration. The probe cap should be on the probe at all times except when sampling.

FIGURE 5
Installing probe with mortar

FIGURE 6
Installed probe, flush with slab

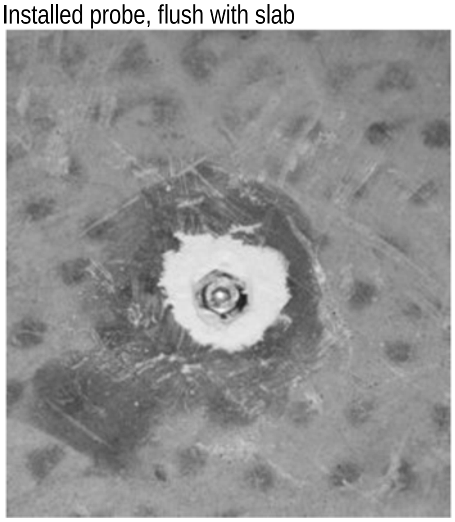


FIGURE 7
Installed probe with sample tube

7.0 Subslab Soil Gas Sampling System Set-Up Procedure

7.1 For semi-permanent and permanent subslab probes, remove the probe cap and attach the sampling union to the subslab probe. Then attach 1/4-inch Teflon® tubing to the sampling union with a Swagelok® nut and ferrule set. See Figure 7.

7.2 Place the helium leak-check enclosure over the subslab probe by threading the Teflon® tubing through the hole of the enclosure. Slide the enclosure down so it seals on the concrete slab. Attach the other end of the sample tube to the sampling manifold with the use of a nut and ferrule set. See Figures 8 - 10.

FIGURE 8
Installing the helium leak check assembly

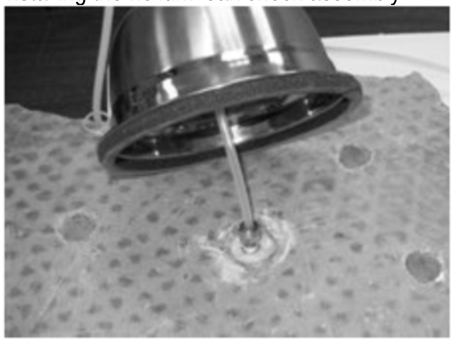
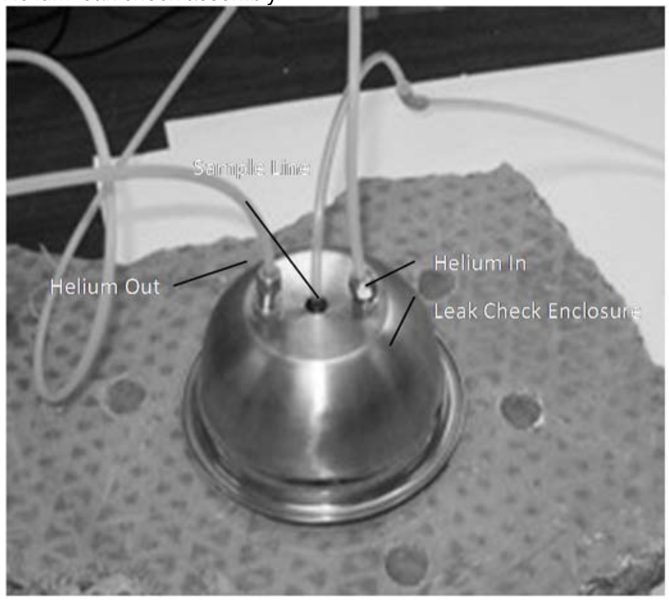


FIGURE 9
Helium leak check assembly



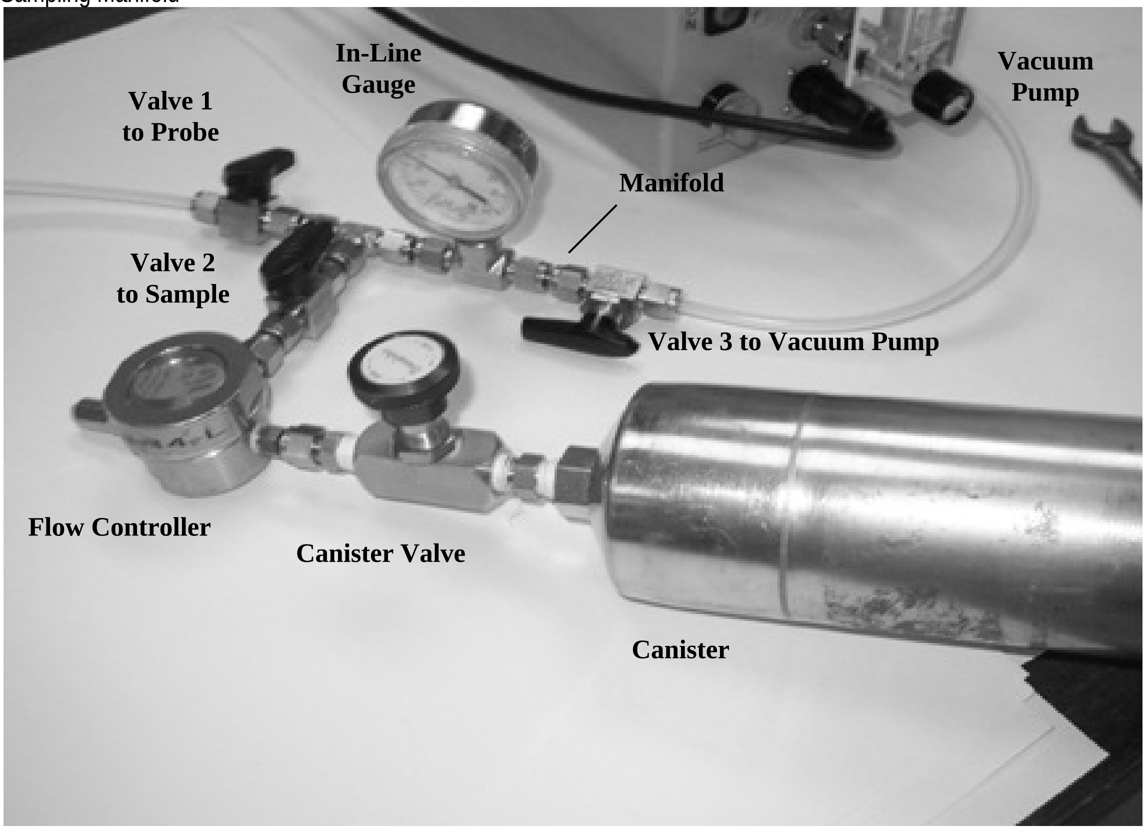
7.3 Attach the subslab sample tubing to the sampling manifold. See Figure 10. Do not connect the canister at this time.

7.3.1 If the sample will be collected over a period of time greater than 30 minutes a flow diversion valve (Swagelok® part# SS-4P4T) should be placed in-line between the probe and the manifold. Once purging has been completed, the flow diversion valve can be turned to the off position, allowing disconnection of the manifold and vacuum pump for use at another location, without the loss of purge integrity at the purged location.

7.3.2 Adjust the vacuum pump to achieve the desired flow rate of 200 mL/min. This should be performed at the outlet of the vacuum pump before purging, either by using a suitable flow meter or calculating the amount of time required to fill a 1-liter Tedlar® bag.

7.4 Attach the air pump to the sampling manifold and the Tedlar® bag to the air pump exhaust.

FIGURE 10
Sampling Manifold



8.0 Subslab Soil Gas Sampling System Leak Checking and Purging Procedure

8.1 Physical Leak Check - Perform a leak check of the sample manifold system (Figure 10):

8.1.1 Make sure the gas probe valve (valve #1) is closed and the sample valve (valve #2) is open.

8.1.2 Open the purge valve (valve #3) and start the vacuum pump. Verify that the flow is set to 200 ml/min.

8.1.3 Close the sample valve (valve #2) and achieve a vacuum gauge reading of 10 inches of mercury (inches Hg) or to a vacuum that will be encountered during sampling, whichever is greater.

8.1.4 A leak-free system will be evident by closing off the purge valve (valve #3), turning off the vacuum pump, and observing no loss of vacuum within the sampling manifold system for a period of 30 seconds. Repair any leaks prior to sample collection by tightening the fittings on the manifold. Re-test to make the sure the manifold passes the physical leak check before proceeding.

8.1.5 Record the leak check date and time on the Subslab Soil Gas Sampling Log.

8.2 System Purge and Helium Leak Check - A purge of the subslab soil gas probe and sampling manifold system is required. The helium leak-check procedure is also performed during this step. This leak check will verify the integrity of the probe seal. This is accomplished by doing the following:

8.2.1 Place the helium leak-check enclosure around the subslab probe to achieve a buildup of helium in the leak-check enclosure. The enclosure should not be tightly sealed and there should be an exhaust for the helium so pressure doesn't build up in the enclosure.

8.2.2 Start the flow of helium to the leak-check enclosure at 200 mL/min. Let the helium fill the enclosure for 1 minute.

8.2.3 Open the sample valve (valve #2) and the purge valve (valve #3) and start the purge pump. Verify that the flow rate is still 200 mL/min.

8.2.4 To start the soil gas probe purge, open the gas probe valve (valve #1) and close the sample valve (valve #2) at the same time, and start timing. It is important to switch these two valves simultaneously. Otherwise, a vacuum can be built up in the sampling system, and its sudden release can draw concrete powder (left at the bottom of the probe hole after drilling) into the sampling system, which will damage the valves and vacuum pump.

8.2.5 If there is shallow groundwater in the area, carefully watch the tubing as the pump is turned on. If water is observed in the sample tubing, shut the pump off immediately. Subslab soil gas collection will not be feasible if the probe is in contact with water.

8.2.6 Connect the helium detector to the enclosure exhaust to confirm that helium is present in the enclosure during purging. It is optional to measure the helium concentration within the enclosure (see Step 7.2.7). Make sure that the helium detector is exposed to ambient air and "zeros out" before measuring the purged soil gas in Step 7.2.7.

8.2.7 Purge the first 30 seconds (approx. 100 mL) into a 1-liter Tedlar® bag. Remove the bag and replace with a fresh 1-L Tedlar® bag. Continue the purge for at least another 2.5 minutes. This will result in a total of about 500 mL of purge gas in the second bag and 600 mL of purge volume total. At the end of the purge time, remove the Tedlar® bag from the pump and connect it to the helium detector. The helium concentration in the purged soil gas must be less than 1 percent of what it was in the helium enclosure during purging to pass the leak test (10,000 parts per million by volume [ppmv] if the helium concentration was 100%) (verify that this limit is consistent with appropriate project-specific regulatory guidance). Either: 1) calculate what 1 percent of the helium concentration was in the enclosure from the measured concentration in Step 7.2.6; or 2) use a limit of 0.1 percent (1,000 ppmv) which allows for a 10-times safety margin. If the probe fails the leak check then corrective action is required.

8.2.8 There are three corrective action options:

8.2.8.1 Make sure that all the fittings are tight and add Teflon tape to them.

8.2.8.2 Try fortifying the probe seal by adding more sealing material or modeling clay and repeating the purge and leak check procedure.

8.2.8.3 If the above two options fail, abandon the hole, drill a new one, and repeat the whole procedure.

Note: **Helium leak detectors may be sensitive to high concentrations of methane or other atmospheric gasses.** If these are expected to be present in the subslab soil gas, then caution should be used with this technique, as false positive readings may be encountered during leak testing. Use a GEM2000® landfill gas meter to determine if methane is present in subslab soil gas.

8.2.9 At the end of the purge and after the system is verified to be leak-free, close the purge valve (valve #3). Do not open it again. Doing so will result in loss of the purge integrity and will require re-purging. Turn off the helium leak detector.

8.2.10 The purged subslab soil gas in the Tedlar® bag can be screened with a GEM2000® landfill gas meter to get field measurements of CO_2, O_2, and CH_4 and/or a MiniRae® PID can be used to measure concentrations of total VOCs in the field.

8.2.11 Record the purge and leak check information on the Subslab Soil Gas Sampling Log.

8.2.12 Immediately move on to the sampling phase. Little to no delay should occur between purging and sampling.

9.0 Subslab Soil Gas Sample Collection Procedure

9.1 Clean sampling protocols must be followed when handling and collecting samples. This requires care in the shipping, storage, and use of sampling equipment. The cleanliness of personnel who come in contact with the sampling equipment is also important, so smoking, eating, drinking, wearing of perfumes or deodorants, and dry-cleaned clothing are prohibited. Canisters should not be transported in vehicles with gas-powered equipment or fuel cans. Sharpie®-type markers should not be used for labeling or note-taking during sampling.

9.2 The air sampling canisters are certified clean and evacuated by the laboratory to ~29 to 30 inches Hg vacuum. Initial canister vacuums that are less than certified by the laboratory are a potential indication of leakage that could affect the accuracy of analytical results. Care should be used at all times to prevent inadvertent loss of canister vacuum. Never open the canister's valve unless the intent is to collect a sample or check the canister vacuum with an attached gauge.

9.3 Verify that the canister has sufficient initial vacuum for sampling. Measure the initial canister vacuum using an external vacuum gauge as described below:

9.3.1 Remove the protective cap from the valve on the canister.

9.3.2 Attach an external gauge, attach the gauge to the canister and open the valve. If the vacuum gauge has two openings, make sure that the other opening is closed; the canister cap can be used for this. After taking the reading, close the canister and remove the gauge.

9.3.3 Measure the initial canister pressure using a digital vacuum gauge with 0.25% accuracy at the -30 to 0 inches Hg range and NIST-traceable calibration for vacuum measurements. See the *Technical Bulletin: Use of External Vacuum Gauges with Canisters* for a recommended model of vacuum gauge[1] for use with Summa canisters used for vapor intrusion sampling.

[1] A PG5 Digital Pressure Gauge from Automation Products Group (APG), Inc.(http://www.apgsensors.com/products/pressure-sensors/digital-pressure-gauges/pg5) with National Institute of Standards and Technology (NIST)-traceable calibration certificate, or equivalent, is recommended for making vacuum measurements.

9.3.4 Do not sample using a canister without sufficient initial vacuum. Be advised that sampling data may be flagged or rejected from canisters with low initial vacuum (less than 28 inches Hg). Low initial vacuum could create a low bias in analytical results due to air leakage. While there is a also a smaller risk that air leakage could introduce contaminants into the canister, the primary concern is the low bias to analytical results; this bias is within the range of analytical variability allowed with the EPA Method TO-15 (±30%) for initial vacuums >24 inches Hg. The table presented in Paragraph 9.3.5 identifies the field team's response based on the initial vacuum reading for a canister. In addition, this table also identifies the potential bias to results at different initial canister vacuums.

9.3.5 Use the following table to determine when to use canisters based on initial vacuum readings.

Initial Vacuum Reading	Potential Error in Analytical Results Due to Leakage	Field Team Response
>30 to 28 inches Hg	Up to -10% error	Use canister for sampling – no limitations on use.
>26 to 28 inches Hg	Up to -21% error	Use canister for sampling if necessary; replace canister with a spare if spares are available.
>24 to 26 inches Hg	Up to -30% error	Sampling with canister is not advisable. Contact project manager and obtain direction before sampling with this canister. Be advised that qualifiers may be applied to analytical results sampled with canisters with vacuums less than 26 inches Hg.
<24 inches Hg	>-30% error	Do not use this canister for sampling. Analytical results will be rejected.

9.4 Attach the canister to the flow controller and then connect the flow controller to the sample valve (valve #2) on the sampling manifold. Open the sample valve (valve #2).

9.5 Before taking the sample, confirm that the sampling system valves are set as follows: (1) the purge valve (valve #3) is confirmed to be closed, (2) gas probe valve (valve #1) is open, and (3) the sample valve (valve #2) is open.

9.6 Slowly open (counter-clockwise) the canister's valve approximately one full turn.

9.7 After sampling for the appropriate amount of time (determined from project instructions; see Table 1), close the sample valve (valve #2) and the canister's valve. If the canister has a built-in or assigned vacuum gauge, allow the canister to fill until the vacuum reaches 2 to 10 inches Hg for 6-liter canisters and 2 to 5 inches Hg for 1-liter canisters. Remove the canister from the sampling manifold.

9.7.1 If sampling for extended periods of time (e.g., 8- or 24-hours), check the samples at some point several hours before the expected completion time (e.g., at 18 or 20 hours for a 24-hour sample) to make sure the canister is collecting at the expected rate. It may also be a good idea to check the canister several hours into the sampling period (e.g., 2 or 4 hours for a 24-hour sample). The flow controllers are rarely set to the exact sampling period.

9.8 If using an external vacuum gauge, re-attach it, open the canister valve, and record the final vacuum. Close the valve, remove the gauge, and replace and tighten the cap on the canister. Ideal final vacuum in the canister is between 2 and 10 inches Hg. More than 10 inches Hg of vacuum following samping can greatly increase reporting limits; however, a small amount of vacuum should be left in the canister so the laboratory can confirm that the canister was not opened during shipment.

9.9 Consult with the project manager before submitting the sample to the laboratory if a final vacuum greater than 10 inches Hg, or less than 2 inches Hg are encountered. Use the following table for guidance to determine how to address final vacuum measurements:

Final Vacuum Reading	Field Team Response
< 2 inches Hg	Contact Project Manager before submitting sample. Notify analytical laboratory to report their laboratory-measured pressure and to get direction from the Project Manager before analyzing sample.
> 2 inches Hg and <10 inches Hg	Submit sample for analysis - no limitations on data use
>10 inches Hg	Contact Project Manager before submitting sample. Verify final vacuum with the analytical laboratory before analysis.

9.10 Canisters with no vacuum left (i.e., 0 inches Hg) should not be analyzed. Contact the Project Manager before submitting a sample with a final vacuum of 0 inches Hg to determine the appropriate course of action. One option is to verify the final vacuum with the analytical laboratory. If there is vacuum remaining in the canister according to the laboratory vacuum gauge, the Project Manager may direct the analytical laboratory to analyze the sample.

9.11 The analytical laboratory should be directed to not analyze a sample showing a final vacuum of 0 inches Hg (as measured by the laboratory), and to notify the Project Manager and obtain further guidance regarding that sample.

9.12 Record the sampling date, times, canister identification (ID), flow controller ID, vacuum gauge ID(s), and any other observations pertinent to the sampling event on the Subslab Soil Gas Sampling Log. Also record the weather conditions (temperature, barometric pressure, precipitation, etc.) during sampling.

9.13 Fill out all appropriate documentation (sampling forms, sample labels, chain of custody, sample tags, etc.).

9.14 Disassemble the sampling system.

9.15 For permanent probes, replace the probe cap and make sure it is securely in place. Cover the probe with duct tape to ensure nobody tampers with it.

9.16 Evacuate the Tedlar® bags outside of the building.

10.0 Altitude Correction

10.1 Air pressure decreases with elevation. Therefore, a canister evacuated at a laboratory located at sea level will show a lower vacuum measurement at a higher altitude. Generally, a 1,000 foot rise in elevation corresponds to a 1 inch Hg drop in pressure OR a 1 inch Hg decrease in measured vacuum. For example, a canister evacuated to 30 inches at sea level and used at 3,000 ft would show an initial vacuum of 27 inches Hg.

10.2 If you plan to sample at altitude, be sure to inform the laboratory ahead of time so they adjust the flow controllers accordingly.

10.3 If sampling is being conducted at higher elevations, verify the elevation difference between the analytical laboratory and field location and determine the associated decrease in measured vacuum.

10.3.1 Calculate the pressure difference between the laboratory and field location as follows: Difference from Sea Level (field) – Difference from Sea Level (laboratory). Use the Altitude Correction Table attached to this SOP.

10.3.2 Subtract the pressure difference determined in Section 10.3.1 from allowable initial vacuum levels (Section 9.3.5) and final vacuum levels (Section 9.9) to determine appropriate initial and final vacuum levels.

11.0 Sample Handling and Shipping Procedure

11.1 Fill out all appropriate documentation (chain of custody, sample tags) and return canisters and equipment to the laboratory

11.2 The canisters should be shipped back to the laboratory in the same shipping container in which they were received. The samples should not be cooled during shipment. DO NOT put ice in the shipping container.

11.3 When packing the canisters for shipment, verify that the valve (just past finger-tight) and valve caps are snug (1/4 turn past finger tight), and use sufficient clean packing to prevent the valves from rubbing against any hard surfaces. Never pack the canisters with other objects or materials that could cause them to be punctured or damaged. Ensure that flow controllers and gauges are separately and adequately wrapped to prevent damage.

11.4 **Do not place sticky labels or tape on any surface of the canister.**

11.5 Place a custody seal over the openings to the shipping container.

11.6 Make sure to insure the package for the value of the sample containers and flow controllers if corporate card policy does not cover this.

11.7 Ship canisters for overnight delivery. NOTE: If sampling on a Friday, ensure the laboratory accepts samples on Saturdays (you do not want the canisters sitting on some loading dock [or worse] for 3 days).

12.0 Subslab Soil Gas Probe Abandonment and Removal Procedure

12.1 After sampling, it is critical that the probe either be removed or securely plugged to prevent the creation of a new pathway for vapor intrusion.

12.2 To remove a temporary probe simply pull on the tubing until the beeswax comes out of the hole.

12.3 To remove a semi-permanent or permanent probe, insert the removal fitting or sampling union into the probe. Using a crowbar, remove the entire probe assembly. If this does not work, use a hammer and chisel to remove the concrete and losen the probe. If the probe cannot be removed in this manner, then over-drill the probe with a rotary hammer drill and 1-inch drill bit.

12.4 Fill the hole with portland cement mix and return the surface as near to pre-sampling conditions as possible.

13.0 Quality Control

13.1 Laboratories supplying canisters must follow the performance criteria and quality assurance prescribed in U.S. Environmental Protection Agency (EPA) Method TO-14/15 for canister cleaning, certification of cleanliness, and leak checking. SOPs are required.

13.2 Laboratories supplying flow controllers must follow the performance criteria and quality assurance prescribed in EPA Method TO-14/15 for flow controller cleaning and adjustment. SOPs are required.

14.0 Attachments

14.1 ***Subslab Soil Gas Probe Installation and Sampling Log - Canister Method***

14.2 ***Subslab Soil Gas Sampling Log - Canister Method***

14.3 ***Sample sign for posting***

14.4 ***Altitude correction table***

Altitude Correction Table

Elevation (ft)	Pressure (in Hg)	Difference from Sea-Level (in Hg)	Elevation (ft)	Pressure (in Hg)	Difference from Sea-Level (in Hg)
0	29.92	0	1500	28.37	1.553
50	29.87	0.053	1550	28.32	1.603
100	29.81	0.106	1600	28.27	1.653
150	29.76	0.159	1650	28.22	1.703
200	29.71	0.212	1700	28.17	1.753
250	29.66	0.265	1750	28.12	1.803
300	29.60	0.317	1800	28.07	1.853
350	29.55	0.370	1850	28.02	1.903
400	29.50	0.422	1900	27.97	1.953
450	29.45	0.474	1950	27.92	2.002
500	29.39	0.527	2000	27.87	2.052
550	29.34	0.579	2050	27.82	2.101
600	29.29	0.631	2100	27.77	2.151
650	29.24	0.683	2150	27.72	2.200
700	29.19	0.735	2200	27.67	2.249
750	29.13	0.787	2250	27.62	2.298
800	29.08	0.838	2300	27.57	2.347
850	29.03	0.890	2350	27.52	2.396
900	28.98	0.941	2400	27.47	2.445
950	28.93	0.993	2450	27.43	2.494
1000	28.88	1.044	2500	27.38	2.543
1050	28.82	1.095	2550	27.33	2.591
1100	28.77	1.147	2600	27.28	2.640
1150	28.72	1.198	2650	27.23	2.688
1200	28.67	1.249	2700	27.18	2.736
1250	28.62	1.299	2750	27.14	2.785
1300	28.57	1.350	2800	27.09	2.833
1350	28.52	1.401	2850	27.04	2.881
1400	28.47	1.452	2900	26.99	2.929
1450	28.42	1.502	2950	26.94	2.977
			3000	26.90	3.025

Note: use the following equation to calculate atmospheric for altitudes not shown on this table:

$P = P_o \exp(-35.523 \times 10^{-6} y)$, where P is the pressure at the desired elevation, P_o is the atmospheric pressure at sea level, and y is the desired elevation. Source: NASA, 1996. *Elevation Correction Factor for Absolute Pressure Measurements*. NASA Technical Memorandum 107240.

Standard Operating Procedure for Indoor, Outdoor, and Crawl Space Air Sampling for VOCs Using Canisters

Quality Management

SOP NO. : AIR SAMPLING-1
ORIGINAL VERSION 06/25/2008 BY BFT
REVISION 1 - 01/17/2011 BY KAS
REVISION 2 - 12/16/2011 BY JNS
REVISION 3 – 6/14/2012 BY JAL

PAGES: 9

File Name: SOP_VI_Air_Sampling_Canister_06142012.Doc

Location: Vapor Intrusion SharePoint Site/Best Practices and OPs/Standard Operating Procedures (SOPs)

Primary Contact(s): Jennifer Simms/PHL and Ben Thompson/CVO

QA/QC Review: Loren Lund/DEN, Shirley Steinmacher/SLC, and Kim Stokes/DFW

Associated Internal Resources:

Technical Bulletins that provided supplemental information can be found on the Vapor Intrusion SharePoint site.

Excel sampling logs are available at Vapor Intrusion SharePoint Site/Best Practices and OPs/Standard Operating Procedures (SOPs).

Example project reports using this SOP can be found at VI Practitioners Site (Limited Access)/Project Document Library

For vapor intrusion evaluations in residential areas, a community outreach plan should be developed and the field team should be trained on how to interact with residents. Internal *Public Involvement Specialists* who can be consulted for this include Amy Brand/VBO and Ginny Farris/WDC.

*** THIS PAGE IS FOR INTERNAL USE ONLY.
DO NOT DISTRIBUTE TO EXTERNAL PARTIES ***

Revision Log

REVISION 3 – 06/14/2012: 1) added specification for a calibrated digital vacuum gauge; 2) revised language for measuring and interpreting initial field vacuum measurements; 3) revised language for measuring and interpreting final field vacuum measurements; 4) added requirement for posting an informational sign on canisters being left unattended; 5) revised language and a revised table for estimating initial and final vacuums at different elevations.

** THIS PAGE IS FOR INTERNAL USE ONLY.
DO NOT DISTRIBUTE TO EXTERNAL PARTIES ***

This SOP is to be used in conjunction with a work plan developed specifically for each project. Please obtain appropriate senior review before implementing this SOP in the field.

Standard Operating Procedure for Indoor, Outdoor, and Crawl Space Air Sampling for VOCs Using Canisters

1. Scope and Application

This standard operating procedure (SOP) describes the approach for collecting indoor, outdoor and crawl space air samples for targeted volatile organic compounds (VOCs). Reporting limits for these samples are usually very low and extremely prone to positive bias from interfering VOC sources. The method presented here is based on 'clean' sampling techniques. The requirements of clean sampling dictate that sampling and sample handling are done by trained personnel. A building survey must be performed before sample collection. It is the responsibility of the project team to make sure this procedure meets all applicable regulatory standards and receives approval/concurrence from the leading regulatory agency for the project. Vapor intrusion (VI) subject-matter experts (SMEs) should be consulted as needed to address technical, regulatory or field implementation issues associated with the use of this SOP.

2. Project Specific Considerations

2.1. Selection of sample locations - Indoor, outdoor and crawl space air sample locations should be selected during the building survey and in consultation with the building owner/occupant. The sample locations should be selected to meet the project-specific data quality objectives. Procedures for performing a building survey are described in the Standard Operating Procedure – Building Surveys for Vapor Intrusion Evaluation.

2.1.1. Guidelines for selecting indoor air sample locations

2.1.1.1. Typically, indoor air samples should be collected from each compartment or heating, air-conditioning, and ventilation (HVAC) zone within a building.

2.1.1.2. Typically, indoor air samples should be collected on the lowest floor of the building at breathing zone height (approximately 3 to 5 feet) toward the center of the building away from windows.

2.1.1.3. Consideration should be given on a case-specific basis to those situations (such as a daycare facility) where a different sampling height may also be appropriate to evaluate a unique setting or population.

2.1.1.4. Indoor air samples should be located in the areas of the building that are occupied most frequently and by the most amount of people.

2.1.1.5. Indoor air samples can be collected from more than one floor within a structure to address varying risk exposures and as part of the process to distinguish contaminants related to vapor intrusion from background sources. Thus, the location and position of the sample container will vary depending on which floor the sampling event takes place.

2.1.1.6. The basement sample(s) are primarily designed to investigate worst-case situations within a structure. Therefore, basement samples are positioned as close as possible to the source area (e.g., sumps or major cracks in the foundation).

2.1.2. Guidelines for selecting outdoor air sample locations

2.1.2.1. Typically, outdoor air samples are collected upwind and/or downwind of the building or site being investigated.

2.1.2.2. Avoid biasing the sample results by placing the canister near potential outdoor VOC sources such as busy roads or gas stations.

2.1.2.3. Outdoor air samples are typically located at least 10 feet away from buildings. However, the outdoor air canister may be placed near the outdoor air intake for the HVAC system for the building.

2.1.2.4. Outdoor air sample canisters should be secured to an immovable structure to ensure security for sampling in public areas. A bicycle lock or piece of chain and padlock can be used. NOTE: Do not secure the canister to or close to a living tree, however, because the tree's evapotranspiration process may release VOCs from groundwater into the vicinity. It may be a good idea to attach a label to the canister explaining that it is an environmental sample and should not be tampered with. The label can also include contact information.

2.1.2.5. Typically, outdoor air samples should be collected at breathing zone height (approximately 3 to 5 feet).

2.1.3. Guidelines for selecting crawl space air sample locations

2.1.3.1. Crawl space air samples are typically collected in locations selected to achieve adequate spatial coverage of the building's crawl space. Sample location selection will be limited by accessibility.

2.1.3.2. Crawl space air sample inlets should be located several feet from the opening or access point to avoid dilution by outdoor air. In cases where the crawl space is most conveniently sampled by access through crawl space vents, a sampling probe (sample delivery line made of Teflon® or stainless steel) of sufficient length is attached to the inlet of the flow controller.

2.2. Selection of sampling duration - Sample collection can be integrated over time by adjusting the flow controller. Project-specific sample periods as short as 10 minutes to as long as 24 hours can be achieved based on the size of canister used and the sampling rate selected (see Table 1). Generally, 6-liter canisters are used for air sampling. The sampling duration is usually selected to mimic the building occupants daily exposure period. Residential air sampling durations are typically 24 hours and commercial/industrial durations are typically 8-hours. However, depending on the workers or occupants schedule this may be adjusted.

TABLE 1
Common Sampling Rates for Air Sampling

Can Size	Length of Sampling Time	Sampling Flow Rate (mL/min)
6 Liter	1 hour	90
6 Liter	8 hours	11.25
6 Liter	24 hours	3.75
1 Liter	5 minutes	180
1 Liter	1 hour	15
850 ml	5 minutes	150
850 ml	1 hour	12

2.3. Selection of sampling schedule - Sample collection should ideally occur during typical operating conditions (i.e., if workers occupy the building from 8am to 4pm, the sample collection would also take place from 8am to 4pm). However, building owners/occupants may request that sampling take place when the building is not in use. In this case, make sure the HVAC system is set to typical operating conditions. Also, consider when the sample pressure will need to be checked (e.g., it's not a good idea to start 24-hour samples at 8am because they will need to be checked around 4am the next day).

3. Health and Safety

There are several health and safety topics to consider when performing air sampling.

3.1. Field teams should work in pairs at residential buildings or at industrial/commercial buildings where a relationship with the building occupant has not yet been established. A field team member should never enter a building alone for the first time. The mental stability of a building occupant should not be taken for granted. Building surveys at abandoned buildings should also be performed in pairs; if one team member is injured, the other will be able to seek help.

3.2. Beware of animals and insects. This applies to abandoned buildings and residences.

3.3. Be careful of overhead hazards in basements. Do not attempt to enter crawl spaces.

3.4. Beware of pinch points and use the correct hand tools to avoid hand injuries.

4. Canister Security

4.1. Field teams should assure that sampling canisters are not disturbed by building occupants.

4.2. If there is a community outreach program associated with the VI sampling event, then information should be made available to building occupants prior to the sampling event that informs occupants about the sampling activities and sampling equipment.

4.3. Each sampling canister should be clearly marked with a sign that includes contact information for a point of contact An example of a sign that can be attached to each sampling canister is provided in the attachment to this SOP. This sign can be edited with project-specific information, laminated and attached to each sampling canister using cable ties (do not attach the signs using adhesive tape).

5. Apparatus and Materials

5.1. Canister, stainless steel, polished, certified clean and evacuated. (Canisters are typically cleaned, evacuated, and provided by the laboratory.)

5.2. Flow controller, certified clean, and set at desired sampling rate. (Flow controllers are typically cleaned, set, and provided by the laboratory.)

5.3. Shipping container suitable for protection of canister during shipping. Typically, strong cardboard boxes are used for canister shipment. The canisters should be shipped back to the laboratory in the same shipping container in which they were received.

5.4. Wrenches and screwdriver (clean and free of contaminants), various sizes as needed for connecting fittings and making adjustment to the flow controller. A 9/16-inch wrench fits the 1/4-inch Swagelok® fittings, which most canisters and flow controllers have.

5.5. Negative pressure (vacuum) gauge, oil-free and clean, to check canister vacuum. (The gauges are typically provided by the laboratory.) The laboratory may either provide one vacuum gauge to be used with all of the canisters, or a vacuum gauge for each canister to be left on during sample collection. Sometimes the canisters are fitted with built-in vacuum gauges that are not removable. These gauges are for field use only, and are an approximate measure of the actual vacuum. Regularly calibrated -- and less rugged -- vacuum gauges are used at the laboratory to measure vacuum before shipment and again after sample receipt.

5.6. Sampling probe, new Teflon® or stainless steel tubing, fitted with compression fittings. (For crawl space samples)

5.7. Swagelok® nut and ferrule set (part #SS-400-NFSET) to connect tubing to the canister

5.8. Sampling cane or similar device for outdoor air sampling to prevent water from entering canister during sampling.

5.9. Shipping container, suitable for protection of canister(s) during shipping. Typically, strong cardboard boxes are used for canister shipment. The canisters should be shipped to the laboratory in the same shipping container(s) in which they were received.

6. Sample Collection Procedure

6.1. Clean sampling protocols must be followed when handling and collecting air samples. This requires care in the shipping, storage, and use of sampling equipment. Cleanliness of personnel who come in contact with the sampling equipment is also important: no smoking, eating, drinking, perfumes, deodorants, dry cleaned clothing, etc. Canisters should not be transported in vehicles with gas-powered equipment or fuel cans. Sharpie®-type markers should not be used for labeling or note-taking during sampling.

6.2. The field team should order some additional canisters in case these are needed to replace visibly damaged canisters or canisters that have leaked during initial leak testing (see Paragraph 6.6.4.

6.3. The sampling canisters are certified clean and evacuated by the laboratory to negative 30 inches mercury (inches Hg). Care should be used at all times to prevent inadvertent loss of canister vacuum. Never open the canister's valve unless the intent is to collect a sample or check the canister vacuum with an attached gauge.

6.4. Prior to taking air samples, be sure to complete a building survey for vapor intrusion evaluations (see SOP – *Building Surveys for Vapor Intrusion Evaluation*). Note any changes in building conditions (especially potential VOC sources) since the building survey was performed.

6.5. Inspect the canister for damage and do not use a canister that has visible damage.

6.6. Verify that the canister has sufficient initial vacuum for sampling. Initial canister vacuums that are less than certified by the laboratory (~29 to 30 inches Hg) are a potential indication of leakage which could affect the accuracy of analytical results. Measure the initial canister vacuum using an external vacuum gauge, as described below:

6.6.1. Remove the protective cap from the canister; make sure the canister valve is closed before doing this.

6.6.2. Attach an external vacuum gauge to the canister and open the valve. If the vacuum gauge has two openings, make sure that the other opening is closed; the canister cap can be used for this. After taking the reading, record the initial vacuum, close the canister valve and remove the gauge.

6.6.3. Measure the initial canister pressure using a digital vacuum gauge with 0.25% accuracy at the -30 to 0 inches Hg range and NIST-traceable calibration for vacuum measurements. See the *Technical Bulletin: Use of External Vacuum Gauges with Canisters* for a recommended model of vacuum gauge[1] for use with Summa canisters used for vapor intrusion sampling.

---

[1] A PG5 Digital Pressure Gauge from Automation Products Group (APG), Inc.(http://www.apgsensors.com/products/pressure-sensors/digital-pressure-gauges/pg5) with National Institute of Standards and Technology (NIST)-traceable calibration certificate, or equivalent, is recommended for making vacuum measurements.

6.6.4. Do not sample using a canister without sufficient initial vacuum. Be advised that sampling data may be flagged or rejected from canisters with low initial vacuum (less than 28 inches Hg). Low initial vacuum could create a low bias in analytical results due to air leakage. While there is a also a smaller risk that air leakage could introduce contaminants into the canister, the primary concern is the low bias to analytical results; this bias is within the range of analytical variability allowed with the EPA Method TO-15 (±30%) for initial vacuums >24 inches Hg. The table presented in Paragraph 6.5.5 identifies the field team's response based on the initial vacuum reading for a canister. In addition, this table also identifies the potential bias to results at different initial canister vacuums.

6.6.5. Use the following table to determine when to use canisters based on initial vacuum readings:

Initial Vacuum Reading	Potential Error in Analytical Results Due to Leakage	Field Team Response
>30 to 28 inches Hg	Up to -10% error	Use canister for sampling – no limitations on use.
>26 to 28 inches Hg	Up to -21% error	Use canister for sampling if necessary; replace canister with a spare if spares are available.
>24 to 26 inches Hg	Up to -30% error	Sampling with canister is not advisable. Contact project manager and obtain direction before sampling with this canister. Be advised that qualifiers may be applied to analytical results sampled with canisters with vacuums less than 26 inches Hg.
<24 inches Hg	>-30% error	Do not use this canister for sampling. Analytical results will be rejected.

6.7. Flow controllers should come pre-set by the laboratory to sample at a pre-determined rate based on specific project requirements (see Table 1 for the most common options). In some cases (that is, project-specific quality assurance [QA]), the flow rate will need to be verified in the field prior to use. This is accomplished with a bubble meter, vacuum source, and instructions supplied by the laboratory.

6.8. In the field log record the canister identification (ID), flow controller ID, initial vacuum, desired flow rate, sample location information, and all other information pertinent to the sampling effort. The indoor and outdoor temperature and barometric pressure should be recorded when sampling begins and is completed.

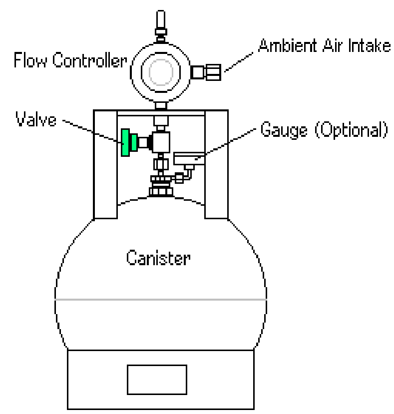
6.9. Connect the flow controller to the canister (Figure 1).

6.9.1. The flow controller fitting denoted "LP" or "OUT" is connected to the canister. Tighten the fitting to be leak free but do not over-tighten (a 1/4 turn past snug is usually enough.) When tightening the fitting, be sure that the valve assembly does not rotate, by using your other hand to hold the valve steady.

6.9.2. If an assigned vacuum gauge is used for each canister, the vacuum gauge should be attached to the canister first and then the flow controller should be attached to the vacuum gauge.

6.9.3. When the flow controller and vacuum gauge are attached correctly they will not move separately from the canister (they will not spin around).

FIGURE 1
Assembled Canister Sampler for Integrated Sample Collection



6.10. For outdoor samples, be sure that the inlet to the flow controller is protected from precipitation. Either place the canister and flow controller under a shelter/enclosure, use a sampling cane provided by the laboratory, or use a clean piece of aluminum foil to build a tent over the flow controller inlet.

6.11. If crawl spaces are being sampled remotely through a crawl space vent, adjust the length of the sampling probe to achieve the desired sampling location and place an inert spacer (wire clothes hanger) near the end of the probe to keep the probe tip opening suspended ~ 3 inches above the ground level. Now connect the sampling probe to the inlet of the flow controller.

6.12. Remove all work articles from the sampling area.

6.13. To begin sampling, slowly open the canister valve one full turn.

6.14. For canisters with built-in or assigned vacuum gauges, monitor the vacuum change several times during the course of the selected sample period to ensure the canister is filling at the desired rate.

6.15. At the end of the sample period, close the canister valve finger tight.

6.16. Remove the flow controller and replace the protective cap on the canister valve fitting.

6.17. Measure the final canister vacuum with the digital vacuum gauge. Attach the digital vacuum gauge, open the canister valve, and record the final vacuum. Close the valve, remove the gauge, and replace and tighten the cap on the canister.

6.18. Ideal final vacuum in the canister is between 2 and 10 inches Hg. More than 10 inches Hg means that a smaller than expected sample volume has been collected, which can increase reporting limits. A small amount of vacuum should be left in the canister to assess the potential for leakage during transport to the laboratory.

6.19. Consult with the project manager before submitting the sample to the laboratory if a final vacuum greater than 10 inches Hg, or less than 2 inches Hg are encountered. Use the following table for guidance to determine how to address final vacuum measurements:

Final Vacuum Reading	Field Team Response
< 2 inches Hg	Contact Project Manager before submitting sample. Notify analytical laboratory to report their laboratory-measured pressure and to get direction from the Project Manager before analyzing sample.
> 2 inches Hg and <10 inches Hg	Submit sample for analysis - no limitations on data use
>10 inches Hg	Contact Project Manager before submitting sample. Verify final vacuum with the analytical laboratory before analysis.

6.20. Canisters with no vacuum left (i.e., 0 inches Hg) should not be analyzed. Contact the Project Manager before submitting a sample with a final vacuum of 0 inches Hg to determine the appropriate course of action. One option is to verify the final vacuum with the analytical laboratory. If there is vacuum remaining in the canister according to the laboratory vacuum gauge, the Project Manager may direct the analytical laboratory to analyze the sample.

6.21. The analytical laboratory should be directed to not analyze a sample showing a final vacuum of 0 inches Hg (as measured by the laboratory), and to notify the Project Manager and obtain further guidance regarding that sample.

6.22. If the flow controller is going to be used for more than one sample collection, be sure to purge it between uses. To do this, attach the flow controller to a vacuum source and draw clean air or gas (ultra-high purity) through it for several minutes before attaching it to the canister.

7. Altitude Correction

7.1. Air pressure decreases with elevation. Therefore, a canister evacuated at a laboratory located at sea level will show a lower vacuum measurement at a higher altitude. Generally, a 1,000 foot rise in elevation corresponds to a 1 inch Hg drop in pressure OR a 1 inch Hg decrease in measured vacuum. For example, a canister evacuated to 30 inches at sea level and used at 3,000 ft would show an initial vacuum of 27 inches Hg.

7.2. If you plan to sample at altitude, be sure to inform the laboratory ahead of time so they adjust the flow controllers accordingly

7.3. If sampling is being conducted at higher elevations, verify the elevation difference between the analytical laboratory and field location and determine the associated decrease in measured vacuum.

7.3.1. Calculate the pressure difference between the laboratory and field location as follows: Difference from Sea Level (field) – Difference from Sea Level (laboratory). Use the Altitude Correction Table attached to this SOP.

7.3.2. Subtract the pressure difference determined in Section 7.2.1 from allowable initial vacuum levels (Section 5.5.4) and final vacuum levels (Section 5.18) to determine appropriate initial and final vacuum levels.

8. Sample Handling and Shipping

8.1. Fill out all appropriate documentation (chain of custody, sample tags) and return canisters and equipment to the laboratory

8.2. The canisters should be shipped back to the laboratory in the same shipping container in which they were received. The samples should not be cooled during shipment. DO NOT put ice in the shipping container.

8.3. When packing the canisters for shipment, verify that the valve (just past finger-tight) and valve caps are snug (1/4 turn past finger tight), and use sufficient clean packing to prevent the valves from rubbing against any hard surfaces. Never pack the canisters with other objects or materials that could cause them to be punctured or damaged. Ensure that flow controllers and gauges are separately and adequately wrapped to prevent damage.

8.4. **Do not place sticky labels or tape on any surface of the canister.**

8.5. Place a custody seal over the openings of the shipping container.

8.6. Make sure to insure the package for the value of the sample containers and flow controllers if corporate card policy does not cover this.

8.7. Ship canisters via overnight delivery. NOTE: If sampling on a Friday, ensure the laboratory accepts samples on Saturdays (you do not want the canisters sitting on a loading dock [or worse] for 3 days).

9. Quality Control

9.1. Canisters supplied by the laboratory must follow the performance criteria and quality assurance prescribed in U.S. Environmental Protection Agency (EPA) Method TO-14/15 for canister cleaning, certification of cleanliness, and leak checking. SOPs are required.

9.2. Flow controllers supplied by the laboratory must follow the performance criteria and QA prescribed in EPA Method TO-14/15 for flow controller cleaning and adjustment. SOPs are required.

10. Attachments

10.1. Indoor, Outdoor, and Crawl Space Air Sampling Log - Canister Method (2 options)

10.2. Sample sign for posting.

10.3. Altitude correction table.

Altitude Correction Table

Elevation (ft)	Pressure (in Hg)	Difference from Sea-Level (in Hg)	Elevation (ft)	Pressure (in Hg)	Difference from Sea-Level (in Hg)
0	29.92	0	1500	28.37	1.553
50	29.87	0.053	1550	28.32	1.603
100	29.81	0.106	1600	28.27	1.653
150	29.76	0.159	1650	28.22	1.703
200	29.71	0.212	1700	28.17	1.753
250	29.66	0.265	1750	28.12	1.803
300	29.60	0.317	1800	28.07	1.853
350	29.55	0.370	1850	28.02	1.903
400	29.50	0.422	1900	27.97	1.953
450	29.45	0.474	1950	27.92	2.002
500	29.39	0.527	2000	27.87	2.052
550	29.34	0.579	2050	27.82	2.101
600	29.29	0.631	2100	27.77	2.151
650	29.24	0.683	2150	27.72	2.200
700	29.19	0.735	2200	27.67	2.249
750	29.13	0.787	2250	27.62	2.298
800	29.08	0.838	2300	27.57	2.347
850	29.03	0.890	2350	27.52	2.396
900	28.98	0.941	2400	27.47	2.445
950	28.93	0.993	2450	27.43	2.494
1000	28.88	1.044	2500	27.38	2.543
1050	28.82	1.095	2550	27.33	2.591
1100	28.77	1.147	2600	27.28	2.640
1150	28.72	1.198	2650	27.23	2.688
1200	28.67	1.249	2700	27.18	2.736
1250	28.62	1.299	2750	27.14	2.785
1300	28.57	1.350	2800	27.09	2.833
1350	28.52	1.401	2850	27.04	2.881
1400	28.47	1.452	2900	26.99	2.929
1450	28.42	1.502	2950	26.94	2.977
			3000	26.90	3.025

Note: use the following equation to calculate atmospheric for altitudes not shown on this table:

$P = P_o \exp(-35.523 \times 10^{-6} y)$, where P is the pressure at the desired elevation, P_o is the atmospheric pressure at sea level, and y is the desired elevation. Source: NASA, 1996. *Elevation Correction Factor for Absolute Pressure Measurements*. NASA Technical Memorandum 107240.

Attachment C
UFP-QAPP

Draft

UFP-Quality Assurance Project Plan for 2013/2014 Vapor Intrusion Sampling, Quanta Resources Corporation Superfund Site, OU1, Edgewater, New Jersey

Prepared for

Honeywell International Inc.

September 2013

CH2MHILL®

DOCUMENT CONTROL NUMBER: CH2M-QUANTA-QAPP-V1
PREPARED USING 2012 USEPA GUIDANCE (HTTP://WWW.EPA.GOV/FEDFAC/PDF/UFP_QAPP_WORKSHEETS.PDF)

Contents

Acronyms and Abbreviations

-	not applicable
%R	percent recovery
$\mu g/m^3$	microgram per cubic meter
AFCEE	Air Force Center for Engineering and the Environment
ARAR	Applicable or Relevant and Appropriate Requirement
BERA	baseline ecological risk assessments
BFB	
CA	corrective action
CAR	Corrective Action Request
CCV	continuing calibration verification
CLP	
CoC	chain of custody
COI	constituents of interest
DFTPP	decafluorotriphenylphosphine
DNAPL	dense nonaqueous phase liquid
DoD	Department of Defense
DQE	Data Quality Evaluation
DQI	data quality indicator
DQO	data quality objective
EB	equipment blank
EDD	electronic data deliverable
FD	Field Duplicate
FS	feasibility study
ft bgs	feet below ground surface
ft^2	square feet
FTL	Field Team Leader
GC/MS	gas chromatography/mass spectrometry
GW	groundwater
HAZWOPER	Hazardous Waste Operations and Emergency Response
HCAA	High-concentration Arsenic Area
Honeywell	Honeywell International Inc.
HTS	Honeywell Tracking Systems
HVAC	heating, ventilation, and air conditioning
IASL	Indoor Air Screening Level
ICAL	initial calibration
ICV	initial calibration verification
IS	internal standards
LCL	lower control limit
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LD	laboratory duplicate
LTM	long-term monitoring
MB	method blank
MDL	method detection limit
mg/kg	milligrams per kilogram (
MIBK	methyl isobutyl ketone
MPC	measurement performance criteria
MRL	method reporting limit

MS	matrix spike
MSD	matrix spike duplicate
NAPL	nonaqueous phase liquid
ND	not detected
NELAC	National Environmental Laboratory Accreditation Conference
NJDEP	New Jersey Department of Environmental Protection
NZ	NAPL zone
OSHA	Occupational Safety and Health Administration
OU1	Operable Unit 1
OU2	Operable Unit 2
PAL	Project Action Limit
PCB	polychlorinated biphenyl
PDF	portable document format
PDI	Pre-Design Investigation
PDWP	Pre-Design Work Plan
PID	photoionization detector
PM	Project Manager
POC	point of contact
PQL	Project Quantitation Limit
PW	
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
QM	Quality Manager
RAO	Remedial Action Objective
RI	remedial investigation
RL	reporting limit
ROD	Record of Decision
RPD	relative percent difference
RPM	Remediation Project Manager
RRF	
RSD	relative standard deviation
RT	retention time
SB	
SDG	Sample Delivery Group
SI	Supplemental Investigation
Site	Quanta Resources Corporation Superfund Site
SOP	Standard Operating Procedures
SRI	Supplemental Remediation Investigation
SRM	standard reference material
SS	
SVOC	semivolatile organic compound
SW	
TBD	to be determined
TPH	total petroleum hydrocarbons
UCL	upper control limit
URL	uniform resource locator
USEPA	U.S. Environmental Protection Agency
VI	vapor intrusion
VOC	volatile organic compound

QAPP Worksheets #1 and #2: Title and Approval Page

1. Project Identifying Information
   a. Site Name/Project Name: Quanta Resources Corporation Superfund Site, Operable Unit 1(OU1)
   b. Site Location/Number: Edgewater, New Jersey
   c. Contract/Work Assignment Number: NJD000606442
2. Lead Organization
   a. Lead Organization Project Manager (PM): Steve Zarlinski/CH2M HILL

      Signature: ______________________ Date: ______________

   b. Lead Organization Quality Manager (QM): Kyle Block/CH2M HILL

      Signature: ______________________ Date: ______________

3. Federal Regulatory Agency: Richard Ho/ U.S. Environmental Protection Agency (USEPA) Region 2 Remedial Project Officer

   Signature: ______________________ Date: ______________

4. State Regulatory Agency: Erica Bergman/ New Jersey Department of Environmental Protection (NJDEP), PM

   Signature: ______________________ Date: ______________

5. Other Stakeholders (as needed):

   Steve Coladonato/Honeywell International Inc. (Honeywell), Site Remediation Manager

   Signature: ______________________ Date: ______________

   Additional Personnel

   Signature: ______________________ Date: ______________

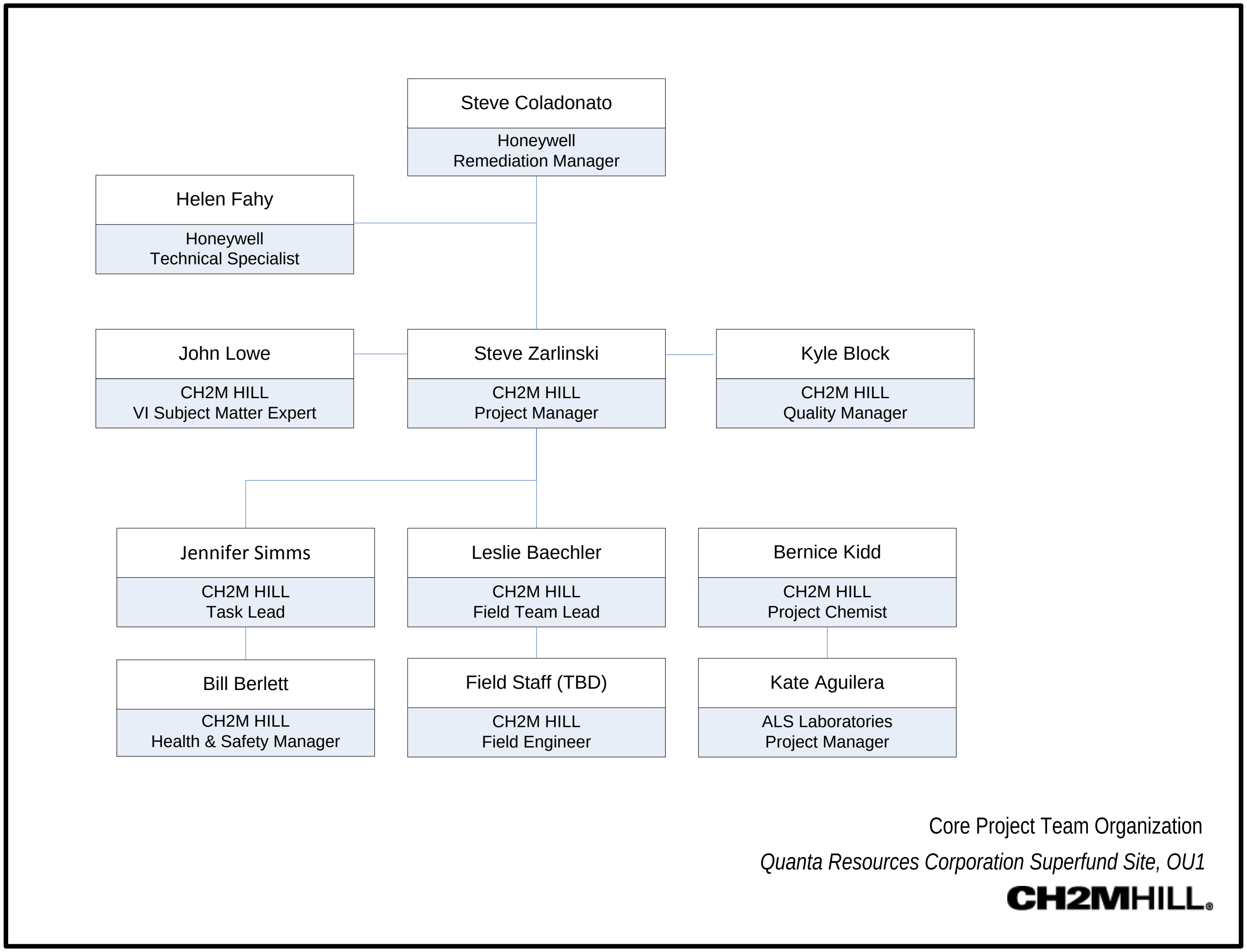
6. List plans and reports from previous investigations relevant to this project

Title	Approval Date
QAPP (Revised), Operable Unit 1, Quanta Resources OU1	October 2005
QAPP (Revised), Operable Unit 1, Quanta Resources OU1	November 2006
QAPP (Revised), Operable Unit 1, Quanta Resources OU1	July 2008
QAPP (Revised), Operable Unit 1, Quanta Resources OU1	October 2011
Quality Assurance Project Plan, Quanta Resources Corporation Superfund Site, Operable Unit 1, Edgewater, New Jersey. August 2012.	August 2012

Note: QAPP - Quality Assurance Project Plan

QAPP Worksheets #3 and #5: Project Organization and QAPP Distribution

(EPA 2106-G-05 Section 2.2.3 and 2.2.4)



QAPP Worksheets #4, #7, and #8: Personnel Qualifications and Sign-off Sheet

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date*
Organization: USEPA				
Richard Ho	Region 2 RPM			
Organization: NJDEP				
Erica Bergman	Remediation PM			
Organization: Honeywell				
Steve Coladonato	RPM			
Helen Fahy	Technical Specialist			
Organization: CH2M HILL				
Steve Zarlinski	CH2M HILL PM			
Kyle Block	QM			
Leslie Baechler	Field Manager			
Jennifer Simms	Task Manager			
John Lowe	Senior VI Specialist			
Bill Berlett	Health and Safety Manager			
Berney Kidd	Project Chemist			

Notes:
*Signatures indicate personnel have read and agree to implement this QAPP as written.
RPM - Remediation Project Manager
VI - vapor intrusion

This project will require uniquely trained personnel to perform specialized field reconnaissance, sampling, onsite/offsite analysis, data review, and other project functions. All project personnel are qualified and experienced in the project tasks for which they are responsible. All field personnel will have received the required Occupational Safety and Health Administration (OSHA) 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) certification and the annual 8-hour refresher course, if applicable. Training records/certificates will maintained by the CH2M HILL.

In addition to the aforementioned roles, subcontractors will be involved on this project to complete the required work, including analytical laboratories and field services subcontractors. The following table provides a list of current subcontractors and their contact phone numbers. Should this list change, an updated list will be provided to the personnel listed in Section 2.3 of this QAPP.

Subcontractor	Role	Contact Name	Telephone Number
ALS Environmental	Analytical Laboratory	Kate Aguilera	805-526-7161, x234

QAPP Worksheet #6: Communication Pathways

Communication pathways will follow the organizational chart provided in Worksheet #3. The Honeywell RPM (Steve Coladonato) and the CH2M HILL PM (Stephen Zarlinski) are the main contacts for questions or concerns. Frequent and timely team communication is important and has a direct connection to the successful delivery of the remedial design. Individual tasks are listed in Worksheets #4, #7, and #8.

Communication Driver	Organization	Name	Contact Information
Regulatory Agency Interface	USEPA Region 2	Richard Ho	212-637-4372, Ho.Richard@epamail.epa.gov
Regulatory Agency Interface	NJDEP	Erica Bergman	609-292-7406, Erica.Bergman@dep.state.nj.us
All project-related tasks	Honeywell	Steve Coladonato	302-791-6738, Steven.Coladonato@Honeywell.com
All project-related tasks	CH2M HILL	Steve Zarlinski	810-360-2061, Steve.Zarlinski@ch2m.com
All project-related tasks	CH2M HILL	Kyle Block	617-626-7013, Kyle.block@ch2m.com
Analytical Actions	CH2M HILL	Berney Kidd	530-229-3203, Bernice.Kidd@CH2M.com
Analytical Actions	ALS Environmental	Kate Aguilera	805-526-7161, ext. 234, Kate.Aguilera@alsglobal.com

QAPP Worksheet #9: Project Planning Session Summary

Date of planning session: May 1, 2013

Location: Conference Call

Purpose: 2013 Vapor Intrusion Strategy Meeting

Participants:

Name	Organization	Title/Role	Phone/Email
Steve Coladonato	Honeywell	RPM	302-791-6738, Steven.Coladonato@Honeywell.com
Steve Zarlinski	CH2M HILL	PM	810-360-2061, Steve.Zarlinski@ch2m.com
Kyle Block	CH2M HILL	QM	617-626-7013, Kyle.block@ch2m.com
Jennifer Simms	CH2M HILL	Task Manager	215-640-9071, ext. 49071, jsimms@ch2m.com
John Lowe	CH2M HILL	Senior VI Specialist	509-464-7325, ext. 27325, jlowe@ch2m.com

QAPP Worksheet #10: Conceptual Site Model

This QAPP addresses sampling and analysis associated with vapor intrusion (VI) sampling at the Quanta Resources Corporation Superfund Site (the Site). The following subsections present the conceptual site model for OU1.

Site Description

The Site is located at the intersection of River Road and Gorge Road in Edgewater, New Jersey. A vacant lot referred to as the Quanta property, portions of River Road and Gorge Road, and properties and portions of properties surrounding the Quanta property comprise the upland portion of the Site, or OU1. The area of OU1 is approximately 24 acres. The Site is adjacent to the Hudson River. Figure 1 depicts the properties constituting OU1.

The Quanta property contains the following:

- Exposed former tank and building foundations
- Remnants of a former oil-water separator
- A wooden bulkhead along the shoreline
- Remains of wooden docks

The property is generally flat and at a lower elevation than River Road to the west and the City Place property to the north. The Quanta, 115 River Road, and i.Park properties are all at a similar elevation, one corresponding to the land surface during industrial use (prior to 1982). River Road and City Place are located at higher elevations due to more recent placement of fill material.

Site History

The brief site history described in this section was provided within the Record of Decision (ROD).

The Site is located at River Road and the intersection of Gorge Road, Edgewater, New Jersey. At the center of the Site is a 5.5-acre vacant lot, referred to as the Quanta property, surrounded by a number of developed and undeveloped parcels, portions of which are also considered part of the Site. Sections of River and Gorge Roads were constructed on top of the Site. The land portion of the Site is approximately 24 acres. The Site also includes an area of sediment contamination in the Hudson River. The extent of the Hudson River sediments affected by the Site will be determined as part of an ongoing Operable Unit 2 (OU2) Remedial Investigation and Feasibility Study (RI/FS) that is separate from this response action.

The Site is characterized by contamination from a variety of industries that operated there from at least the 1870s to 1981. These industries included coal tar processing, chemical manufacturing, and waste oil storage. The 5.5-acre Quanta property is a remnant of an industrial coal tar facility that once covered approximately 15 acres.

Source Areas

Soil and groundwater at OU1 contain constituents from industries that operated on various portions of the Site from at least the 1870s to 1981, including coal tar processing, chemical manufacturing, and waste oil storage. A substantial amount of data and information was collected and assessed during the OU1 RI and various supplemental investigations, and has achieved the objective of completing a comprehensive site characterization of the Site. The RI and various supplemental investigations included the following:

- Characterization of OU1 sources
- Determination of the nature and extent of contamination
- Evaluation of fate and transport of constituents of interest (COIs)
- Assessment of potential risks to human health and the environment

Soils at OU1 have been found to contain source material in the form of principal threat and low-level threat waste. Principal threat wastes are considered source materials; that is, materials that include or contain hazardous substances, pollutants, or contaminants that act as a reservoir for migration of contamination to groundwater, surface water, or as a source for direct exposure. Principal threat wastes are those source materials considered to be highly toxic or highly mobile that generally cannot be reliably contained or would present a significant risk to human health or the environment should exposure occur.

Nonaqueous Phase Liquid

The location, nature, and extent of the nonaqueous phase liquid (NAPL) at OU1 have been comprehensively delineated through the RI process for the purposes of remedial design and implementation. NAPL is found throughout the Site and is made up of aromatic volatile organic compounds (VOCs) and semivolatile organic compounds (SVOCs). Most of the SVOCs are polycyclic aromatic hydrocarbons. Coal tar materials found at the Site range from low-viscosity liquids to solid tar. NAPL at the Site is identified as either free-phase or residual NAPL. Free-phase NAPL is able to collect in monitoring wells; whereas, residual NAPL is present in quantities insufficient to allow mobility. Most of the free-phase NAPL at the Site is denser than water and is present in six discrete areas (NAPL zones [NZs] 1 through 6), shown in Figure 1. These NZs were defined on the basis of one or more of following factors:

- Thickness of visually observed NAPL, as well as TarGOST response indicating NAPL
- Lateral and vertical separation between areas where visual observations and TarGOST results indicated significant thicknesses of NAPL
- Observation of free-phase (that is, mobile) NAPL in monitoring wells

All principal threat NAPL at the Site (that is, the portion of the NAPL for which active remediation is required according to the ROD) is present within the boundaries of these six zones (Figure 1). Exposure through direct contact, ingestion, or inhalation is plausible for NZ-1 and NZ-2, and the potential is likely that future use of the Site could result in exposure if appropriate remedial actions are not implemented. Direct exposure to NAPL in NZ-3, NZ-4, or NZ-6 is unlikely, even under a construction scenario, given their depth (generally deeper than 10 feet below ground surface [ft bgs]). Without additional remedial effort, there is the potential for the migration of free-phase NAPL to sediment and surface water in the Hudson River from NZ-2 and NZ-5. As indicated in the ROD, release of free-phase NAPL from NZ-1, NZ-3, NZ-4, and NZ-6 to sediments is not plausible.

Outside the defined NZs and throughout the Site, NAPL is present as residual NAPL or thin, discontinuous pockets of free-phase NAPL that have not been found to be contiguous with the defined NZs. These areas contain constituents exceeding soil remediation values but are not identified as source areas; therefore, they “can generally be managed in place with engineering controls (capping) and proper land-use restrictions” (according to the ROD).

High-Concentration Arsenic Area

The High-concentration Arsenic Area (HCAA) lies beneath a cover and fill material, and a portion is located beneath an active access ramp to the City Place development. The extent of the HCAA is defined as soil concentrations of arsenic greater than 1,000 milligrams per kilogram (mg/kg). The 1,000-mg/kg boundary (that is, the HCAA) as depicted in Figure 1 was developed using historical data collected during the investigation, remediation, design, and development of the City Place property, and new data collected during the RI and supplemental investigations. The cover placed during construction of the City Place development extends beyond the delineated 1,000-mg/kg boundary along the edge of River Road on the Quanta property. The extent of the HCAA and the extent of the cover are depicted in Figure 1.

Arsenic Areas

Eight separate sources of soluble arsenic to groundwater and of other inorganic constituents in groundwater were identified as principal threat waste within the lateral extent of OU1 but outside the HCAA. Each arsenic area has elevated soil concentrations that have a potential direct-contact risk to human health defined as greater than 390 mg/kg for arsenic contamination in the shallow, unsaturated soils (approximately the first 4 feet of surface soil) and 1,000 mg/kg for deeper soils. The ROD refers to these areas as “shallow arsenic hotspots,” although these areas may contain residual NAPL or overlap areas delineated as NZs (Figure 1). One of these arsenic areas (referred to as AA-3), located on the i.Park property, was excavated in December 2010 by i.Park Edgewater, LLC, with oversight by CH2M HILL. The remaining seven arsenic source areas and the HCAA are depicted in Figure 1.

Site-wide Soil

Outside the areas defined as NZs, residual NAPL, discontinuous pockets of free-phase NAPL, and Site-related constituents are found in saturated and unsaturated soil. The primary Site COIs driving the remediation are VOCs, SVOCs, and metals, particularly arsenic and lead. Remedial evaluations and design are being based on a subset of representative constituents, including:

- VOCs: benzene, ethylbenzene, toluene, and xylenes
- SVOCs: naphthalene and benzo(a)pyrene
- Arsenic
- Lead

Some of the remaining constituents detected less frequently in exceedance of the soil remediation goals will be addressed in source areas through the remedial actions for the source areas. Outside the source areas, capping and institutional controls will meet the remedial action objectives for these other constituents by preventing direct contact.

Site-wide Groundwater

Similar Site-related constituents were detected in groundwater throughout the Site. As noted in the ROD, “groundwater contamination above screening criteria, with similar constituents, has been identified from remedial investigations of all neighboring properties”; therefore, a complete delineation of the extent of groundwater impacts from the Site is not possible. USEPA concluded that the factors listed in Table 1, along with “the presence of off-site sources and regional characteristics…would undermine a successful restoration within OU1.”

TABLE 1
Characteristics Limiting Groundwater Restoration Potential

Type of Factor	Characteristics Limiting Groundwater Restoration Potential
Contaminant	Widespread presence of NAPL (primarily as DNAPL), and recalcitrant DNAPL-related constituents
	Long history of industrial use and associated releases at and outside of the Site
	Volume and depth of contaminated media
	Presence of arsenic in soil and groundwater, and the collocation of arsenic and DNAPL
Hydrogeologic	Complex geology consisting of interbedded and undulating layers of sands, silts, and clays with discontinuous peat deposits
	Heterogeneous soil conditions and the presence of low-permeability materials, such as silts and clays
Site setting	Highly urbanized environment with significant surficial and subsurface infrastructure

Note:
DNAPL - dense non-aqueous-phase liquid

A waiver of groundwater Applicable or Relevant and Appropriate Requirements (ARARs) within the OU1 boundary was granted by USEPA on the basis of technical impracticability from an engineering perspective, and Remedial Action Objectives (RAOs) were developed to prevent unacceptable exposure and limit offsite migration. The complete evaluation of groundwater restoration potential is documented in the technical impracticability evaluation (CH2M HILL, 2010).

Vapor Intrusion Sampling

VI investigation activities at the Site began in 2006 as part of the OU1 remedial investigation, and VI investigation and monitoring has continued annually since then at the 115 River Road, 163 Old River Road, and 103 River Road buildings. The investigation and monitoring activities have been conducted in accordance with work plans approved by the USEPA and the NJDEP. The ROD and Administrative Order of Consent for OU1 summarized the conclusions of the VI investigation and identified continuing activities for addressing VI during implementation of remedial action at the Site. As detailed within the ROD, results of the VI investigation and monitoring events since 2006 indicate that the VI pathway has not caused indoor air concentrations to exceed USEPA's guidelines for exposure to indoor air.

Work plans were submitted to USEPA in December 2012 to define the specific long-term monitoring (LTM) activities in the three buildings during remedial action at OU1. LTM activities identified in these plans included: annual monitoring at the 115 River Road building until it underwent demolition as required to perform remedial action; monitoring conducted every 5 years at the restaurant building; and discontinuing monitoring at the Medical Arts building unless changing conditions warranted resumed monitoring. Following agency comments submitted in February 2013, discussions of LTM approaches were deferred, and work plans were submitted to the agencies describing monitoring activities only for 2013, which were conducted in March 2013 and detailed in letter report *Quanta Resources Corporation Superfund Site, Operable Unit 1 (OU1) Vapor Intrusion—Results of the 2013 Monitoring Events at 115 River Road, 163 Old River Road, and 103 River Road,* dated May 21, 2013 (CH2M HILL).

Honeywell met with the agencies on July 23, 2013 to review VI investigation and monitoring results for the Site and to discuss a path forward for VI activities during OU1 remedial design and subsequent remedial action. The outcomes from that meeting are:

- Submitting a letter Work Plan for winter 2013/2014 VI monitoring at the three buildings
- Performing that monitoring during the fourth quarter 2013
- Reporting the results to USEPA and NJDEP

115 River Road (Office/Commercial Use Building)

The main building at 115 River Road is an old manufacturing building that has been converted into 11 different tenant-occupied 'buildings,' which are further divided into multiple commercial/office spaces. Some portions of the building are constructed on top of a crawl space, while others are either slab-on-grade or have a basement, as shown in Figure 1. There is an interim VI mitigation measure in place in the Building 7/8 basement and Building 6 crawl space; it consists of a ventilation system that generates approximately 15 indoor-to-outdoor air exchanges per hour. The system is currently operated by the building owner, who keeps it running 24 hours per day. The building owner also performs housekeeping activities within Building 7/8 (for example, keeping covers on the sumps and floor drains) to reduce the potential for VI. CH2M HILL conducts monthly inspections of the ventilation system and basement.

163 Old River Road Building (Tomaso's Restaurant)

The 163 Old River Road building had multiple former uses (for example, quality control [QC] laboratory, storage, and office space) but has since been converted into a restaurant. The building is currently occupied by Tomaso's Restaurant (previously Jono's). The building is approximately 6,000 square feet (ft^2) and consists of two stories with a single-story front dining area.

103 River Road Building (Medical Arts)

The 103 River Road building was constructed in the early 1980s and is two-and-a-half stories, occupying a footprint of approximately 5,000 ft^2 (100-feet long by 50-feet wide). The building contains three separate office spaces: a medical office and a dental office on the first floor, and a cardiologist office on the second floor.

QAPP Worksheet #11: Project/Data Quality Objectives

This QAPP addresses the VI sampling portion of the winter 2013/2014 VI monitoring event for the Site.

VI monitoring is part of a routine monitoring effort being performed at the Site until the final remedy has been implemented in accordance with the ROD and as detailed during the July 23, 2013 meeting between the NJDEP, the USEPA, and Honeywell. The objective of this monitoring is to confirm that the conditions at each building remain the same as documented in 2011 ROD; specifically, the ROD states: although “elevated levels of site contaminants” have been detected in subslab soil gas, “the detected levels [in indoor air] have not exceeded USEPA’s guidelines for exposure to indoor air.”

The historical VI sampling results, along with the winter 2013/2014 indoor air monitoring results, will be reviewed to determine the effectiveness of the VI mitigation system in reducing concentrations in the Building 7/8 basement at the 115 River Road building.

QAPP Worksheet #12: Measurement Performance Criteria

As part of the winter 2013/2014 VI monitoring event, environmental samples will be collected from several matrices. Environmental samples will be required to meet selected performance criteria, as indicated in the following tables. Measurement performance criteria are acceptance limits selected for project-specific sampling and analytical systems that will be used to judge whether project quality objectives are met. These measurement performance criteria should be satisfied in order to support defensible decisions and provide sufficient data for ultimately completing the implementation of the remedial action.

Data quality will be evaluated based on their precision, accuracy, representativeness, completeness, and comparability.

Precision

Precision is a measure of reproducibility of analytical results. It can be defined as the degree of mutual agreement among individual measurements obtained under similar conditions. Total precision is a function of the variability associated with both sampling and analysis. Precision will be evaluated as the relative percent difference (RPD) between field duplicate (FD) sample results and laboratory sample duplicates, or between the matrix spike (MS) and matrix spike duplicate (MSD) results. FDs will compose 10 percent of the sampling effort. MS/MSD samples will be field designated at a 5 percent frequency.

Accuracy

Accuracy is the degree of agreement between a measured value and the ‘true’ (or expected) value. It represents an estimate of total error from a single measurement, including either systematic error (bias) or random error that may reflect variability due to imprecision. Accuracy is evaluated in terms of percent recoveries determined from results of MS/MSD and laboratory control sample (LCS) analyses.

Representativeness

Representativeness is the degree to which sample data accurately reflect the characteristics of a population of samples. It is achieved through a well-designed sampling program and by using standardized sampling strategies and techniques, and analytical procedures. Factors that can affect representativeness include site homogeneity, sample homogeneity at a single point, and available information around which the sampling program is designed. Using multiple methods to measure an analyte can also result in nonrepresentativeness of sample data.

Completeness

Completeness is the amount of valid measurements compared to the total amount generated. It will be determined for each method, matrix, and analyte combination. The completeness goals of each project are optimized to meet the data quality objectives (DQOs). The goals for this program are 95 percent.

Comparability

Comparability is the confidence with which one data set can be compared to another. It is achieved by maintaining standard techniques and procedures for collecting and analyzing samples, and reporting the analytical results in standard units. Results of performance evaluation samples and systems audits will provide additional information for assessing comparability of data among participating subcontractor laboratories.

Matrix: Air

Analytical group or method: Volatile organics, EPA Method TO-15, and TO-15 SIM

Concentration level: Low

DQI	QC Sample or Measurement Performance Activity	Measurement Performance Criteria
Precision	FDs	≤50% RPD for analytes at least 5X reporting limit
Accuracy/Precision (laboratory)	LCS and LCSD	Criteria listed in Table 28-6
Sensitivity	Initial calibration and Form I for all environmental samples	See Worksheet #15-3
Accuracy	Internal Standards	Criteria listed in Table 28-6
Accuracy	Surrogate Compounds	Criteria listed in Table 28-6
Accuracy	Lab MB	< RL

Notes:
DQI - Data Quality Indicator
LCSD – laboratory control sample duplicate
MB – method blank
RL – reporting limit

QAPP Worksheet #13: Secondary Data Criteria and Limitations

Secondary data refer to historical data previously collected for the sites. The source(s) of the data, date of collection, planned uses, and limitations of the secondary data for each site are summarized in the following table.

Data Type	Source	Data Uses Relative to Current Project	Factors Affecting the Reliability of Data and Limitations on Data Use
GW, SW, SS, Sediment, NAPL	Various, historical	Historical data to be used as needed based on limitations	The data is treated as screening-level qualitative measurements, since full analytical QC data was not received with the data, nor was there conclusive indication that full review was conducted.
Soil, GW	CH2M HILL, OU1 RI	To help identify potential source areas and plan sampling locations	No limitation
Soil, GW, SW, Air, NAPL	CH2M HILL, OU1 SI	To help identify potential source areas and plan sampling locations	No limitation
Sediment	CH2M HILL, OU2 sediment study	To help identify potential source areas and plan sampling locations	No limitation
Air	CH2M HILL, VI events	To help identify potential source areas and plan sampling locations	No limitation
Soil, GW, SW, PW	CH2M HILL, 2008–2009 SRI	To help identify potential source areas and plan sampling locations	No limitation
Soil	CH2M HILL, 2008–2009 soil sampling	To help identify potential source areas and plan sampling locations	No limitation
Sediment	CH2M HILL, 2008 OU2 BERA	To help identify potential source areas and plan sampling locations	No limitation
Soil, NAPL	CH2M HILL, 2010 soil sampling	To help identify potential source areas and plan sampling locations	No limitation
Soil, NAPL	CH2M HILL, 2011 soil sampling	To help identify potential source areas and plan sampling locations	No limitation
GW, Soil, Air	CH2M HILL, 2012 PDI	To help identify potential source areas and plan sampling locations	No limitation

Notes:
BERA— baseline ecological risk assessments
GW - groundwater
PCB— polychlorinated biphenyls
PDI— Pre-Design Investigation
PW – porewater
SI— Supplemental Investigation
SRI— Supplemental Remediation Investigation
SS – surface soil
SW – surface water
TPH—total petroleum hydrocarbons

QAPP Worksheets #14 and #16: Project Tasks and Schedule

The work scope involves the following tasks:

- Work Plan Submittal – September 30, 2013
- Procurement of Laboratory – November 30, 2013
- Mobilization for sampling – mid-December 2013
- 2013/2014 VI Sampling Event – 6 days to complete
- Laboratory Results Obtained – mid-January 2014
- Submit Report to USEPA – Spring 2014

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits

The following tables contain the list of target compounds or COIs that will be evaluated for this project. Due to the nature of the project, no Project Action limits (PALs) or Project Quantitation Limits (PQLs) have been specified or are required, with the exception of air monitoring.

Matrix: Indoor Air/Outdoor Air/Crawl Space Air

Analytical method: EPA Method TO-15 and TO-15 SIM

Concentration level (if applicable): Low

TABLE 15-3

Target Parameter		PAL ($\mu g/m^3$)[a]	PQL Goal ($\mu g/m^3$)	Laboratory-specific Quantitation Limit[b] ($\mu g/m^3$)	Estimated Detection Limit[c] ($\mu g/m^3$)
VOCs	Benzene	1.6	<PAL	0.10	0.10
	Ethylbenzene	4.9	<PAL	0.50	0.50
	Naphthalene	0.36	<PAL	0.025	0.025
	Tetrachloroethene	47	<PAL	0.50	0.50
	Trichloroethene	3.0	<PAL	0.50	0.50
	1,2,4-Trimethylbenzene	31	<PAL	0.50	0.50
	1,3,5-Trimethylbenzene	31	<PAL	0.50	0.50
	m&p-Xylenes	440	<PAL	0.50	0.50
	o-Xylenes	440	<PAL	0.50	0.50

Notes:

[a] PALs are based on NJDEP Nonresidential IASLs Tables (March 2013) and USEPA Regional Screening Levels (May 2013) for industrial air, whichever is less.
[b] Quantitation limit equals the laboratory RL.
[c] Detection limit equals the laboratory MDL.

$\mu g/m^3$ – microgram per cubic meter
IASL - Indoor Air Screening Level
MDL – method detection limit

Matrix: Soil Gas

Analytical method: EPA Method TO-15

Concentration level (if applicable): Low

TABLE 15-3

Target Parameter		PAL ($\mu g/m^3$)[a]	PQL Goal ($\mu g/m^3$)	Laboratory-specific Quantitation Limit[b] ($\mu g/m^3$)	Estimated Detection Limit[c] ($\mu g/m^3$)
VOCs	Benzene	16	<PAL	0.10	0.10
	Ethylbenzene	49	<PAL	0.50	0.50
	Naphthalene	3.6	<PAL	0.50	0.50
	Tetrachloroethene	470	<PAL	0.50	0.50
	Trichloroethene	30	<PAL	0.50	0.50
	1,2,4-Trimethylbenzene	310	<PAL	0.50	0.50
	1,3,5-Trimethylbenzene	310	<PAL	0.50	0.50
	m&p-Xylenes	4,400	<PAL	0.50	0.50
	o-Xylenes	4,400	<PAL	0.50	0.50

Notes:

[a] PALs are based on NJDEP Nonresidential IASLs Tables (March 2013) and USEPA Regional Screening Levels (May 2013) for industrial air, whichever is less.
[b] Quantitation limit equals the laboratory RL.
[c] Detection limit equals the laboratory MDL.

QAPP Worksheet #17: Sampling Design and Rationale

Sampling design and rationale were developed to both account for occupied spaces and to accommodate agency comments on select areas.

Sampling at 115 and 103 River Road will be performed during the work week (Monday to Friday) while tenants are occupying the building. Sampling at 163 Old River Road will be performed while the restaurant is closed, sometime between 9 pm Sunday and 5 pm Tuesday, as requested by the building owners and consistent with previous events. The heating, ventilation, and air conditioning (HVAC) system will be set to typical operating conditions, and the building's windows and doors will be kept shut during the sampling event at each building. The field sampling team will check on each building periodically to verify these conditions persist throughout the entire sampling period.

Sampling will not be performed during storm events or within 48 hours of a significant rain event (that is, greater than 1 inch of rain in a 24-hour period) because of the potential influence such conditions may have on indoor air, outdoor air, and subslab soil gas. Information on weather conditions in Edgewater, New Jersey, during the sampling event will be obtained from Weather Underground's Web site.[1]

Sampling Locations

The sampling locations are provided in Worksheet #18 and shown in the figures attached to the VI Work Plan.

- **115 River Road.** The sampling locations were modified from previous years to address comments from NJDEP during the July 2013 and via email (Attachment A of the VI Work Plan). Indoor air sampling will be performed at each of the occupied tenant spaces on the first floor at the 115 River Road building. Indoor air samples will also be collected from the Building 7/8 basement where the ventilation system is operating. Additionally, indoor air samples will be collected from the second and third floor occupied tenant spaces closest to the elevator in Building 8 to assess if the elevator is acting as a preferential vapor pathway. Crawl space air samples will be collected at Buildings 2, 4, and 6.
- **163 Old River Road.** The same locations that were sampled in previous years will be sampled during the winter 2013/2014 VI monitoring event.
- **103 River Road.** The same locations that were sampled in previous years will be sampled during the winter 2013/2014 VI monitoring event. The three existing subslab soil gas probes will be removed and replaced with Cox Colvin vapor pins because two of them are rusted shut due to flooding from Hurricane Sandy.

---

1 http://www.wunderground.com/.

QAPP Worksheet #18: Sampling Locations and Methods

Sampling locations and methods are outlined in the following subsections.

Sample ID	Matrix	Depth (ft bgs)	Type	Analyte/ Analytical Group	Sampling SOP	Comments/Sample Description
115 River Road						
Q1-IA-32	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Building 2 1st floor - main open space
Q1-IA-35	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Building 4 1st floor - Conference room on side table (center of Building 4)
Q1-IA-28	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Building 6 1st floor - storage room on northern side near former stairway
Q1-IA-36	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Building 7 1st floor - far eastern room
Q1-IA-37	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Building 7 1st floor - western room next to stairs
Q1-IA-21	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Building 7/8 basement - hallway near Sump 2
Q1-IA-23	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Building 7/8 basement - far eastern room - middle of room near the floor drain
Q1-IA-25	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Building 7/8 basement - main room near Sump 1
Q1-IA-42	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Building 8 2nd floor - TBD- occupied tenant space near elevator
Q1-IA-43	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Building 8 3rd floor - TBD - occupied tenant space near elevator
Q1-IA_40	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Building 9 1st floor - western side open space near utility room door
Q1-IA-41	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Building 9 1st floor - eastern side storage room
Q1-IA-44	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Building 10 1st floor - Suite 1001 - TBD
Q1-IA-45	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Building 10 1st floor - Suite 1003 - TBD
Q1-IA_46	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Building 10 1st floor - Suite 1027 - TBD
Q1-IA-39	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Building 11 1st floor - western side of main room
Q1-CS-01	Air	-	Crawl Space	VOCs/Method TO-15	SOP #003	Building 6 crawl space - northwestern side
Q1-CS-04	Air	-	Crawl Space	VOCs/Method TO-15	SOP #003	Building 4 crawl space - southern side
Q1-CS-07	Air	-	Crawl Space	VOCs/Method TO-15	SOP #003	Building 2 crawl space - southern side
Q1-OA-03	Air	-	Outdoor	VOCs/Method TO-15	SOP #003	115 River Road southern-most parking lot chained to fence near Building 10
Q1-OA-09	Air	-	Outdoor	VOCs/Method TO-15	SOP #003	South of 115 River Road Building next to Hudson River near Building 1
Q1-OA-10	Air	-	Outdoor	VOCs/Method TO-15	SOP #003	Northwestern corner of Building 12 at Site fence
163 Old River Road						
Q2-IA-01	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Kitchen - counter top
Q2-IA-02	Air	-	Indoor	VOCs/Method TO-15	SOP #003	1st floor dining room - on table near wall

Sample ID	Matrix	Depth (ft bgs)	Type	Analyte/ Analytical Group	Sampling SOP	Comments/Sample Description
Q2-IA-03	Air	-	Indoor	VOCs/Method TO-15	SOP #003	2nd floor dining room - on table in southwestern room
Q2-VI-01	Air	-	Subslab	VOCs/Method TO-15	SOP #002	Storage room next to stairs
Q2-VI-02	Air	-	Subslab	VOCs/Method TO-15	SOP #002	Kitchen - northern side next to water service closet
Q2-OA-01	Air	-	Outdoor	VOCs/Method TO-15	SOP #003	Southern side of 163 Old River Road Building - chained to fence
Q2-OA-02	Air	-	Outdoor	VOCs/Method TO-15	SOP #003	Northwest of parking lot - chained to fence
103 River Road						
Q3-IA-01	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Medical office storage room
Q3-IA-02	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Dentist office hallway by exit door
Q3-IA-03	Air	-	Indoor	VOCs/Method TO-15	SOP #003	Medical office reception area
Q3-VI-01	Air	-	Subslab	VOCs/Method TO-15	SOP #002	Medical office storage room
Q3-VI-02	Air	-	Subslab	VOCs/Method TO-15	SOP #002	Southern stairwell
Q3-VI-03	Air	-	Subslab	VOCs/Method TO-15	SOP #002	Medical office utility room
Q3-OA-01	Air	-	Outdoor	VOCs/Method TO-15	SOP #003	Northern side of 103 River Road building
Q3-OA-02	Air	-	Outdoor	VOCs/Method TO-15	SOP #003	Southwestern corner of the 103 River Road Building

Notes:

Samples may be adjusted based on Site conditions.
Actual number of QC samples (equipment blank, duplicates, and MS/MSD) will be based on total amount of samples collected in accordance with Worksheet #20.

- - not applicable
ID - identification
SOP – standard operating procedure

QAPP Worksheets #19 and #30: Sample Containers, Preservation, and Hold Times

The sampling objectives for analysis by an offsite laboratory are described in this worksheet. The project-specific target analytes and required methods are defined in the following table, along with the appropriate bottleware to be used for each analysis.

Laboratory (name, sample receipt address, point of contact [POC], email, and phone numbers): ALS Environmental, 2655 Park Center Drive, Ste. A, Simi Valley, California 93065, POC: Kate Aguilera, 805-526-7161, ext. 234, Kate.Aguilera@alsglobal.com

List any required accreditations/certifications: Laboratory has current National Environmental Laboratory Accreditation Conference (NELAC) or New Jersey certification.

Back-up Laboratory: None

Sample delivery method: Fed Ex

Analytical Group	Matrix	Method/SOP	Containers (Number, Size, and Type per Sample)	Preservation Requirements	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround
VOCs	Air	TO-15 and TO-15 SIM	6-L Summa Canister	None	-	30 days	21-day
VOCs	Soil gas	TO-15	6-L Summa Canister	None	-	30 days	21-day

QAPP Worksheet #20: Field QC Summary

QC samples will be collected to monitor accuracy, precision, and the presence of field contamination for analytical methods to be performed in the offsite laboratory. The following table outlines the frequency of collection of the QC samples.

Matrix	Analytical Group	Analysis	Field Samples	FDs	MSs	MSDs	Field Blanks	Equipment Blanks	Trip Blanks	Total No. of Samples Analyzed
Air	Indoor	VOCs, TO-15	22	3	-	-	-	-	-	25
Soil gas	Subslab	VOCs, TO-15	5	1	-	-	-	-	-	6
Air	Crawlspace	VOCs, TO-15	3	1	-	-	-	-	-	4
Air	Outdoor	VOCs, TO-15	7	0	-	-	-	-	-	7

Notes:

To assess the representativeness of the sampling methods, FDs will be collected at a minimum frequency of 1 per 10 environmental samples per matrix.

MS/MSD samples (MS/MSD for organics; MS and laboratory duplicate [LD] for inorganics) will be taken at a frequency of 1 pair per 20 field samples. These samples are used to assess the effect of the sample matrix on the recovery of target compounds or target analytes.

QAPP Worksheet #21: Field SOPs

Field SOPs are included as Attachment B to the VI Work Plan.

SOP#	Title, Revision, Date, and URL (if Available)	Originating Organization	SOP Option or Equipment Type (if SOP Provides Different Options)	Modified for Project? Y/N	Comments
001	Installation and Extraction of the Vapor Pin, May 20, 2011	Cox Colvin	-	No	
002	Installing Subslab Probes and Collecting Subslab Soil Gas Samples Using Canisters, June 14, 2012	CH2M HILL	-	No	
003	Indoor, Outdoor, and Crawl Space Air Sampling for VOCs Using Canisters, June 14, 2012	CH2M HILL	-	No	
004	Conducting Building Surveys for VI Evaluations, December 16, 2011	CH2M HILL	-	No	

Note:
URL – uniform resource locator

QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection

Field Equipment	Activity	SOP Reference	Title or Position of Responsible Person	Frequency	Acceptance Criteria	CA
Dielectric MGD-2002, or equivalent	Per Manual	Per Manual	FTL	Per Manual	Per Manual	Per Manual
LandTec GEM2000 Landfill Gas Meter	Per Manual	Per Manual	FTL	Per Manual	Per Manual	Per Manual
PID	Per Manual	Per Manual	FTL	Per Manual	Per Manual	Per Manual

Notes:

CA – corrective action

FTL - Field Team Lead

PID - photoionization detector

QAPP Worksheet #23: Analytical SOPs

Laboratory must have current NELAC or New Jersey certification.

SOP#	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
VOA-TO15	Determination of Volatile Organic Compounds in Air Samples Collected in Specially Prepared Canisters and Gas Collection Bags by Gas Chromatography/Mass Spectrometry (GC/MS): EPA TO-15	Definitive	VOCs in Air	HP 5890/5972 HP 6890/5973 Agilent 6890/5973 Agilent 7890/5973	ALS Environmental, Simi Valley, CA	No

QAPP Worksheet #24: Analytical Instrument Calibration

Identify all analytical instrumentation that requires calibration, and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and CA requirements on the worksheet.

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
GC/MS	Tuning Verification	Once every 24-hours or analytical batch	Ion abundance criteria as described in Table 3 of Method TO-15	1) Repeat BFB analysis 2) Retune instrument	Wida Ang (Dept. Supervisor)	VOA-TO15 _Rev.20
GC/MS	ICAL – minimum of five levels	Initially or if continuing calibration no longer meets criteria	1) <30% RSD with 2 exceptions up to 40% (AFCEE: only ≤30% RSD) 2) Area response at each calibration level within 40% of IS mean area response over the ICAL range 3) RT for each IS within 20s of the mean RT over the ICAL range	1) May repeat 1 point (if 5 levels) or 2 points (if 6 levels) 2) Inspect the system for problems, and perform required maintenance 3) Repeat ICAL Problem must be corrected. Samples may not be analyzed until there is a valid ICAL.	Wida Ang (Dept. Supervisor)	VOA-TO15 _Rev.20
GC/MS	ICV	Following every ICAL	Percent difference of ±30%	Correct problem, and verify second source standard. Rerun second source verification. If that fails, correct problem, and repeat ICAL. Problem must be corrected. Samples may not be analyzed until there is a valid ICV.	Wida Ang (Dept. Supervisor)	VOA-TO15 _Rev.20
GC/MS	CCV	Once every 24 hours, if an ICAL has not been performed (within the last 24 hours)	Percent difference of ±30% Note: If CCV is biased high, and analyte is ND, results are acceptable and will be noted in case narrative	1) Reanalyze CCV 2) Identify and correct problem; re-analyze or, if necessary, qualify the data 3) Repeat ICAL if CCV CA is unsuccessful	Wida Ang (Dept. Supervisor)	VOA-TO15 _Rev.20

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
GC/MS	IS	All samples, duplicates, blanks, and standards	1) RT must be <20 seconds from most recent valid calibration (ICAL midpoint or CCV) 2) Area response ±40% of IS area response of most recent valid calibration (ICAL midpoint or CCV)	1) Identify and correct the problem 2) Reanalyze the sample unless obvious matrix interference exists 3) Problem persists, qualify data	Wida Ang (Dept. Supervisor)	VOA-TO15 _Rev.20
GC/MS	Surrogate Standards	All samples, duplicates, blanks, and standards	70-130% recovery (AFCEE: 60-140%)	1) Identify and correct the problem 2) Reanalyze the sample unless obvious matrix interference exists 3) Problem persists, qualify data	Wida Ang (Dept. Supervisor)	VOA-TO15 _Rev.20
GC/MS	Laboratory MB	Once every analytical batch of 20 or fewer samples	No analyte detected equal to or greater than the MRL (DoD: No analytes > ½ MRL; common lab contaminants, none detected > MRL)	1) Reanalyze blank 2) Identify and correct problem 3) Reanalyze blank and affected samples 4) Qualify data	Wida Ang (Dept. Supervisor)	VOA-TO15 _Rev.20
GC/MS	LCS	Once every analytical batch of 20 or fewer samples	%R within laboratory generated limits (AFCEE, AZ: 70-130% recovery)	1) Reanalyze 2) Identify and correct the problem 3) Qualify data*	Wida Ang (Dept. Supervisor)	VOA-TO15 _Rev.20
GC/MS	LD	Once every analytical batch of 20 or fewer samples	RPD within ±25% for positive hits	1) Analyze third aliquot 2) Flag data if third aliquot unacceptable	Wida Ang (Dept. Supervisor)	VOA-TO15 _Rev.20
GC/MS	Holding Time	-	SUMMA canisters - 30 days (USEPA Region 9 - 14 days) Tedlar bags - 72 hours (not included in AFCEE Manual)	Contact client and qualify data	Wida Ang (Dept. Supervisor)	VOA-TO15 _Rev.20

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
GC/MS	MRL	-	At or greater than the low standard of the current ICAL AFCEE: Minimum 2x MDL; DoD: Minimum 3x MDL	-	Wida Ang (Dept. Supervisor)	VOA-TO15 _Rev.20
GC/MS	MDL with Limit of Detection Verification	Initially and once per 12 month period	Limit of Detection Verification - Response with a minimum signal to noise ratio of 3:1	-	Wida Ang (Dept. Supervisor)	VOA-TO15 _Rev.20
GC/MS	Report results between MDL and MRL	-	Upon request (Required for AFCEE and DoD projects; verify with Client QAPP)	Qualify results as estimated	Wida Ang (Dept. Supervisor)	VOA-TO15 _Rev.20

%R - percent recovery
AFCEE - Air Force Center for Engineering and the Environment
CCV - Continuing Calibration Verification
DoD – Department of Defense
ICAL - Initial Calibration
ICV - Initial Calibration Verification
IS - Internal Standards
MRL - method reporting limit
ND - not detected
RSD - relative standard deviation
RT – retention time

QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person	SOP Reference[1]
GC/MS	Replace septa, clean injection port, clip replace column	TO-15	Leak test, column and injection port inspection, source insulator integrity	Daily or as needed	Passing BFB and CCV, passing IS response	Perform maintenance, check standards, recalibrate	Laboratory Analyst	VOA-TO15 _Rev.20

QAPP Worksheet #26 and #27: Sample Handling, Custody, and Disposal

Sampling organization: CH2M HILL

Laboratory: ALS Environmental

Method of sample delivery (shipper/carrier): Federal Express—Priority Overnight

Number of days from reporting until sample disposal: Minimum 30 days after final report sent to the client

Activity	Organization and Title or Position of Person Responsible for the Activity	SOP Reference
Sample labeling	FTL, CH2M HILL	As Per Work Plan
CoC form completion	FTL, CH2M HILL	As Per Work Plan
Packaging	FTL, CH2M HILL	As Per Work Plan
Shipping coordination	FTL, CH2M HILL	As Per Work Plan
Sample receipt, inspection, and log-in	Kate Aguilera/ALS Environmental	As Per Work Plan
Sample custody and storage	Kate Aguilera/ALS Environmental	As Per Work Plan
Sample disposal	Kate Aguilera/ALS Environmental	As Per Work Plan

Note:
CoC – chain of custody

Collecting data of known quality begins at the point of sample collection. Legally defensible data are generated by adhering to proven evidentiary procedures. These procedures are outlined in the following sections and must be followed to preserve and ensure the integrity of all samples from the time of collection through analysis. Sample custody records must be maintained both in the field and in the subcontractor laboratory. A sample is considered to be in someone's custody if it is either in his or her physical possession or view, locked up, or kept in a secured and restricted area. Until shipment, sample custody will be the responsibility of the sampling team leader.

CoC records document sample collection and shipment to the laboratory. A CoC form will be completed for each sampling event. The original copy will be provided to the laboratory with the sample shipping cooler, and a copy will be retained in the field documentation files. The CoC form will identify the contents of each shipment and maintain the custodial integrity of the samples. All CoC forms will be signed and dated by the responsible sampling team personnel. The "relinquished by" box will be signed by the responsible sampling team personnel, and the date, time, and air bill number will be noted in the CoC form. The laboratory will return the executed copy of the CoC form with the hardcopy report.

The shipping packages containing the samples will be sealed with a custody seal any time they are not in an individual's possession or view before shipping. All custody seals will be signed and dated by the responsible sampling team personnel.

At a minimum, the CoC-custody form must contain:

- Site name
- PM, Project Chemist, and Data Manager names, telephone numbers, and fax numbers
- Unique sample ID
- Date and time of sample collection
- Source of sample (including name, location, sample type, and matrix)
- Number of containers
- Preservative used
- Analyses required
- Name of sampler
- Custody transfer signatures, and dates and times of sample transfer from the field to transporters and to the laboratories
- Bill of lading or transporter tracking number (if applicable)
- Turnaround time
- Lab name, address, and contact information
- Any special instructions

Erroneous entries in CoC records will be corrected by drawing a line through the error and entering the corrected information. The person performing the correction will date and initial each change made in the CoC form.

Laboratory Sample Custody Procedures (receipt of samples, archiving, and disposal): A custodian at the laboratory will accept custody of the shipped samples, and check them for discrepancies, proper preservation, integrity, etc. If noted, issues will be forwarded to the laboratory project or laboratory manager for CA; ALS will be notified and will be involved in CA decisions. The sample custodian will relinquish custody to the appropriate department for analysis. A Condition upon Receipt or similar form will be completed by the laboratory and will be included in the analytical data package.

Disposal of the samples will occur only after analyses and quality assurance (QA)/QC checks are completed, and 60 days after issuance of analytical report.

QAPP Worksheet #28: Analytical Quality Control and Corrective Action

Matrix: Air and soil gas

Analytical group: VOCs

Analytical method/SOP: TO-15

TABLE 28-6

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	CA	Title/Position of Person Responsible for CA	Project-specific MPC[a]
Tuning (Mass calibration check)	At beginning of run and every 24 hours	Meet all DFTPP criteria	Check instrument, reanalyze, re-tune	Lab personnel	Meet all DFTPP criteria
Method Blank (Lab Reagent Blank)	Prior to each analytical run	< RL	Identify source and correct prior to continuing analysis	Lab personnel	< RL
ICAL	Prior to sample analysis	RSD ≤ 30% of average RRF; no more than 10% of analytes failure	Check instrument, recalibrate; qualify data	Lab personnel	RSD ≤ 30% of average RRF; no more than 10% of analytes failure
CCV Standard	Every 12 hours	RRF % D ≤ 30%; not more than 10% of analytes failure	Recalibrate, qualify data	Lab personnel	RRF % D ≤ 30%; not more than 10% of analytes failure
LCS/LCSD	2 per batch of ≤ 20 samples	70 – 130% R; RPD ≤ 20%	Qualify data unless high recovery and/or ND	Lab personnel	70 – 130% R; RPD ≤ 20%
Lab QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits [a.]	CA	Person(s) Responsible for CA	MPC
Surrogate Compounds	All samples, standards, and blanks	70–130% R	Re-inject; qualify data	Lab personnel	70–130% R
IS	All samples, standards, and blanks	-30 to +40% from initial/continuing calibration (for Superfund program)	Check instruments, reanalyze affected samples if possible. If reanalysis not possible qualify data (except NDs with high IS recovery).	Lab personnel	-30 to +40% from initial/continuing calibration

Notes:
[a] Recovery and precision limits and measurement performance criteria are generic.
% D
DFTPP – decafluorotriphenylphosphine
MPC - Measurement Performance Criteria
RRF -

QAPP Worksheet #29: Project Documents and Records

The electronic data will be used to generate validation reports, modeling results, data summary tables, maps, and other figures. This program will follow CH2M HILL standard procedures for environmental data collection.

Data collected during the field demonstrations will be reduced and reviewed, and a report on the findings will be tabulated in a standard format. The criteria used to identify and quantify the analytes will be those specified in the analytical methods. Data deliverables will be reported as "Contract Laboratory Program (CLP)-like" Sample Delivery Groups (SDGs). In addition, the USEPA will have access to the lab and Site data upon request.

The completed copies of the CoC records (both external and internal) accompanying each sample from time of initial bottle preparation to completion of analysis will be attached to the analytical reports.

The Data Package will consist of SDGs in portable document format (PDF)". The laboratory will upload the electronic data deliverable (EDD) directly to the project LocusFocus EIM database. The laboratory will provide the data package deliverables 21 business days after sample receipt of a complete SDG. The Project Chemist will check the package to make sure all deliverables are provided. If problems are identified, the laboratory will be alerted, and CAs will be requested. The data will be used to generate summary tables.

The electronic deliverable formats will be submitted according to the Honeywell EDD requirements. All deliverables must also undergo a QC check by the laboratory before delivery. The original data, tabulations, and electronic media are stored in a secure and retrievable fashion.

The PM or Data Manager will maintain close contact with the Project Chemist so that all nonconformance issues are acted upon prior to data manipulation and assessment routines.

Data will be archived in project files and on electronic archive tapes for the duration of the project, 5 years, or as specified in contractual agreements.

The data flow from the laboratory and field to the project staff and data users will be sufficiently documented so that data are properly tracked and reviewed before use.

In addition to the data management procedures, the laboratory will maintain sufficient electronic and hardcopy records to recreate each analytical event. The minimum records the laboratory will keep include the following:

- Raw data, including instrument printouts, bench work sheets, or chromatograms with compound ID and quantitation reports
- Laboratory-specific written SOPs for each analytical method and QA/QC function in place at the time of analysis of project samples
- Record keeping requirements for non-analytical data included in the Pre-Design Work Plan (PDWP)

The following table lists field files and assessment reports that will be kept on file. Additional records will be kept on an as needed basis.

Sample Collection Documents and Records	Onsite Analysis Documents and Records	Offsite Analysis Documents and Records	Data Assessment Documents and Records	Other
• Site and field logbooks • CoC forms • Request forms and associated correspondence	• PID readings • GEM2000 readings	• Internal CoC forms • Sample preparation log • Standard traceability logs • Instrument calibration data • Instrument analysis logs • QC summary checklist with all relevant information • Sample analysis data • Instrument/computer printouts • Definition of qualifiers • Final report	• Sample acceptance checklist • CA reports	• Telephone/email logs • CA documentation • Equipment maintenance logs • Procurement request forms

QAPP Worksheets #31, #32, and #33: Assessments and Corrective Action

Assessments

Assessment Type	Responsible Party and Organization	Number/Frequency	Estimated Dates	Assessment Deliverable	Deliverable Due Date
Health and Safety Audit	Bill Berlett, Health and Safety Manager, CH2M HILL	Minimum of 1 per year	TBD	Audit checklist	TBD
Laboratory Audit	Bernice Kidd, Project Chemist, CH2M HILL	As needed	-	Laboratory Audit Report	As needed

Assessment

QA audits may be performed by or under the direction of the project QM. These audits will be implemented to evaluate the capability and performance of project and subcontractor personnel, items, activities, and documentation of the measurement system(s). The QM may plan, schedule, and approve system and performance audits based upon the CH2M HILL SOP customized to the project requirements. At times, the QM may request additional personnel with specific expertise from company or project groups to assist in conducting performance audits. However, these personnel will not have responsibility for the project work associated with the performance audit.

System Audits

System audits, performed by the QM or designated auditors, will encompass a qualitative evaluation of measurement system components to ascertain their appropriate selection and application. In addition, field and laboratory QC procedures and associated documentation may be system audited. These audits may be performed once during the performance of the project. However, if conditions adverse to quality are detected or if the PM requests, additional audits may occur.

Performance Audits

The laboratory will be required to conduct an analysis of Performance Evaluation samples or provide proof that Performance Evaluation samples submitted by USEPA or a state agency have been analyzed within the past 12 months.

Formal Audits

Formal audits refer to any system or performance audit that is documented and implemented by the QM. These audits encompass documented activities performed by qualified lead auditors to a written procedure or checklists to objectively verify that QA requirements were developed, documented, and instituted in accordance with contractual and project criteria. Formal audits may be performed on project and subcontractor work at various locations.

Auditors who have performed the Site audit after gathering and evaluating all data will write audit reports. Items, activities, and documents determined by lead auditors to be in noncompliance will be identified at exit interviews conducted with the involved management. Noncompliances will be logged and documented through audit findings, which are attached to and are a part of the integral audit report. These audit-finding forms are directed to management to satisfactorily resolve the noncompliance in a specified and timely manner.

The PM has overall responsibility to act upon all CAs necessary to resolve audit findings promptly and satisfactorily. Audit reports must be submitted to the PM within 15 days of completion of the audit. Serious deficiencies will be reported to the PM within 24 hours. All audit checklists, audit reports, audit findings, and acceptable resolutions are approved by the QM prior to issue. Verification of acceptable resolutions may be determined by re-audit or documented surveillance of the item or activity. Upon verification acceptance, the QM will close out the audit report and findings.

Assessment Response and Corrective Action

Assessment Type	Responsibility for Responding to Assessment Findings	Assessment Response Documentation	Timeframe for Response	Responsibility for Implementing CA	Responsible for Monitoring CA Implementation
Health and Safety Audit	Steve Zarlinski, PM CH2M HILL	Letter and any verification documentation	24 hours after notification	FTL, CH2M HILL	Bill Berlett, Health and Safety Manager CH2M HILL

Corrective Action

The following procedures were established so that conditions adverse to quality, such as malfunctions, deficiencies, deviations, and errors, are promptly investigated, documented, evaluated, and corrected. CA forms are included in the PDWP.

Procedure Description

When a significant condition adverse to quality is noted at Site, laboratory, or subcontractor location, the cause of the condition will be determined and CA will be taken to preclude repetition. Condition identification, cause, reference documents, and CA planned to be taken will be documented and reported to the QM, PM, FTL, and involved subcontractor management, at a minimum. Implementation of CA is verified by documented follow-up action.

All project personnel have the responsibility, as part of the normal work duties, to promptly identify, solicit approved correction, and report conditions adverse to quality. CAs will be initiated as follows:

- When predetermined acceptance standards are not attained
- When procedure or data compiled are determined to be deficient
- When equipment or instrumentation is found to be faulty
- When samples and analytical test results are not clearly traceable
- When QA requirements have been violated
- When designated approvals have been circumvented
- As a result of system and performance audits
- As a result of a management assessment
- As a result of laboratory/field comparison studies
- As required by USEPA methods

Project management and staff, such as field investigation teams, remedial response planning personnel, and laboratory groups, monitor ongoing work performance in the normal course of daily responsibilities. Work may be audited at the CH2M HILL office, sites, laboratories, or subcontractor locations. Activities or documents ascertained to be noncompliant with QA requirements will be documented. CAs will be mandated through audit finding sheets attached to the audit report. Audit findings are logged, maintained, and controlled by the Task Manager.

Personnel assigned to QA functions will have the responsibility to issue and control Corrective Action Request (CAR) forms. The CAR identifies the out-of-compliance condition, reference document(s), and recommended CA(s) to be administered. The CAR is issued to the personnel responsible for the affected item or activity. A copy is also submitted to the PM. The individual to whom the CAR is addressed returns the requested response promptly to the QA personnel, affixing his/her signature and date to the CA block, after stating the cause of the conditions and CA to be taken. The QA personnel maintain the log for status of CARs, confirms the adequacy of the intended CA, and verifies implementation. CARs will be retained in the project file for the records.

Any project personnel may identify noncompliance issues; however, the designated QA personnel are responsible for documenting, numbering, logging, and verifying the closeout action. The PM is responsible for implementing, documenting, and approving all recommended CAs.

Reports to Management

CH2M HILL management personnel receive QA reports appropriate to their level of responsibility. The PM receives copies of all QA documentation. QC documentation is retained within the department that generated the product or service, except where this documentation is a deliverable for a specific contract. QC documentation is also submitted to the QM for review and approval. Previous sections detailed the QA activities and the reports that they generate. A final audit report for each project may also be prepared. The reports may include:

- Periodic assessment of measurement data accuracy, precision, and completeness
- Results of performance audits and system audits
- Significant QA problems and recommended solutions for future projects
- Status of solutions to any problems previously identified

Additionally, any incidents requiring CA will be fully documented.

QAPP Worksheet #34: Data Verification and Validation Inputs

Verification Input	Description	Internal/External	Responsible for Verification (Name, Organization)
Field Notes	Field notes will be reviewed internally and placed in the Site file. A copy of the field notes will be attached to the final report.	Internal	Leslie Baechler, FTL, CH2M HILL
CoC and Shipping Forms	CoC and shipping forms will be reviewed internally upon completion and verified against the packed coolers they represent. The shippers' signature in the CoC should be initialed by the reviewer, a copy of the CoC retained in the Site file, and the original and remaining copies placed inside the package for shipment.	Internal	Leslie Baechler, FTL, CH2M HILL
Audit Report	Upon report completion, a copy of all audit reports will be placed in the Site file. If CAs are required, a copy of the documented CA taken will be attached to the appropriate audit report in the Site file. At the beginning of each week and at the completion of the Site work, Site file audit reports will be reviewed internally so that all appropriate CAs have been taken and that CA reports are attached. If CAs have not been taken, the Site Manager will be notified so that no action is taken until CAs are completed.	Internal	Steve Zarlinski, PM, CH2M HILL
Laboratory Data	All laboratory data packages will be verified externally by the laboratory performing the work for completeness and technical accuracy prior to submittal.	External	ALS Environmental (Kate Aguillera)

QAPP Worksheet #35: Data Verification Procedures

Step IIa/IIb	Validation Input	Description	Person(s) Responsible for Validation
IIa	Field SOPs	Verify that the sampling SOPs were followed	Leslie Baechler, FTL, CH2M HILL
IIa	Analytical SOPs	Verify that the analytical SOPs were followed	Laboratory QA Officer/TBD Berney Kidd/CH2M HILL
IIa	Method QC Results	Verify that the required QC samples were run and met required limits	Laboratory QA Officer/TBD Berney Kidd/CH2M HILL
IIa/IIb	Data Validation	Validate 100 percent of the data to confirm quality	Berney Kidd/CH2M HILL
IIa/IIb	Data Usability Evaluation	Evaluate data based on precision, accuracy, representativeness, comparability, and completeness for project objectives	Berney Kidd/CH2M HILL
IIb	Onsite Screening (such as PID readings)	Verify that the field data meets Work Plan requirements for completeness and accuracy based on field calibration records	Leslie Baechler, FTL, CH2M HILL
IIb	Field Documentation	Verify accuarcy and completenss of field notes	Leslie Baechler, FTL, CH2M HILL
IIb	Field QC Sample Results	Verify that the required field QC samples were run and met required limits	Laboratory QA Officer/TBD Berney Kidd/CH2M HILL

QAPP Worksheet #36: Data Validation Procedures

TABLE 36-1
Data Validation Procedures

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (Title and Organizational Affiliation)
IIa/IIb	Air	VOCs	Low	Defined in following text	Berney Kidd/CH2M HILL Peeyush Gupta/HTS

Note:

HTS – Honeywell Tracking Systems

Personnel involved in data validation will be independent of any data generation effort. The Project Chemist will be responsible for overseeing data validation. Data validation will be carried out when the data packages are received from the laboratory. It will be performed on an analytical batch basis using the summary results of calibration and laboratory QC, as well as those of the associated field samples. Data packages will be reviewed for all COIs. Raw data will be reviewed for approximately 10 percent of the data packages or as deemed necessary by the Project Chemist. Validation will be performed using the following procedures and those referenced for Level 3 or 4, as appropriate:

- Review the data set narrative to identify any issues that the lab reported in the data deliverable
- Check sample integrity (sample collection, preservation, and holding times)
- Evaluate basic QC measurements used to assess the accuracy, precision, and representativeness of data, including QC blanks, LCSs, MS/MSDs, surrogate recovery when applicable, and FD or LD results
- Review sample results, target compound lists, and detection limits to verify that project analytical requirements are met
- Initiate CAs, as necessary, based on the data review findings
- Qualify the data using appropriate qualifier flags, as necessary, to reflect data usability limitations

Level 3 validation procedures will also include reviewing the evaluation of calibration and QC summary results against the project requirements and other method-specific QC requirements.

Data validation will be patterned after USEPA guidelines for organic data review, substituting the calibration and QC requirements specified in this QAPP for those specified in the guidelines. The flagging criteria in Tables 36-2 and 36-3 will be used. The qualifier flags are defined in Table 36-4.

Qualifier flags, if required, will be applied to the electronic sample results. If multiple flags are required for a result, the most severe flag will be applied to the electronic result. The hierarchy of flags from the most severe to the least severe is as follows: R, UJ, U, and J.

Any significant data quality problems will be brought to the attention of the Project Chemist.

TABLE 36-2
Flagging Conventions for Organic Methods
Quanta, Quality Assurance Project Plan

Quality Control Check	Evaluation	Flag	Samples Affected
Holding Time	Holding time exceed for extraction or analysis by less than a factor of two	J positive results UJ NDs	Affected samples
	Holding time exceed for extraction or analysis by a factor of two	J positive results R NDs	Affected samples
GC/MS Instrument Performance Check	Mass assignment in error and laboratory cannot reprocess data	R all results	All samples in batch
	Ion abundance criteria not met	R all results if critical ions involved, use judgment otherwise	All samples in batch
ICAL GC/MS Methods	%RSD > 30%	J positive results UJ NDs	Analyte in associated samples
CCV	**% difference or % drift >30% with high recovery**	J positive results	Analyte in associated samples
GC/MS Methods (ICV and CCV)	**% difference or % drift <30% with low recovery**	J positive results UJ NDs	Analyte in associated samples
LCS	%R >UCL	J positive results	Analyte in associated samples
	%R <LCL but ≥10%	J positive results UJ NDs	Analyte in associated samples
	%R <LCL but ≤10%	J positive results R NDs	Analyte in associated samples
MB <RL	Convert blank to soil units if necessary, multiply highest blank value by 5 (by 10 for common lab contaminants, acetone, methylene chloride, MIBK, cyclohexane, phthalates)	U positive results <5 x highest blank concentration (<10 x for common contaminants)	All associated samples in batch
Surrogates	%R >UCL	J positive results	Parent sample
	%R <LCL but ≥10%	J positive results UJ NDs	Parent sample
	%R <LCL but ≤10%	J positive results R NDs	Parent sample

TABLE 36-2
Flagging Conventions for Organic Methods
Quanta, Quality Assurance Project Plan

Quality Control Check	Evaluation	Flag	Samples Affected
Internal Standards -30% to +40% recovery	Area > UCL	J positive results	Associated analytes in sample
	Area < LCL	J positive results UJ NDs	Associated analytes in sample
	Area < 25%	J positive results R NDs	Associated analytes in sample
LDs ± 25% precision	Both sample results >5 times RL and RPD>UCL	J positive results	LD pair
	One or both samples <5 times RL and a difference between results of ± 2 times RL	J positive results UJ NDs	LD pair
FDs ± 30% precision	Both sample results >5 times RL and RPD>UCL	J positive results	FD pair
	One or both samples <5 times RL and a difference between results of ± 2 times RL	J positive results UJ NDs	FD pair

Notes:
ICAL should be based on average response factors or a linear regression equation. Laboratories will need Project Chemist approval to use a nonlinear calibration curve.
LCL – lower control limit
MIBK - methyl isobutyl ketone
UCL – upper control limit

TABLE 36-4
Qualifier Flag Definitions
Quanta, Quality Assurance Project Plan

Flag	Definition
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.
U	This analyte was analyzed for but ND at the specified detection limit.
UJ	The analyte was analyzed for, but was ND. The reported quantitation limit is approximate and may be inaccurate or imprecise.
NJ	The analysis indicates the presence of an analyte that has been 'tentatively identified,' and the associated numerical value represents its approximate concentration.

QAPP Worksheet #37: Data Usability Assessment

The data usability assessment is an evaluation based on the results of data verification and validation in the context of the overall project decisions or objectives. The assessment determines whether the project execution and resulting data meet the project DQOs. Both the sampling and analytical activities must be considered, with the ultimate goal of assessing whether the final, qualified results support the decisions to be made with the data.

All the results will be assembled and statistically reported for an overall Data Quality Evaluation (DQE), which will be provided as an appendix to the VI Monitoring Report. The assessment report will identify precision and accuracy exceedances with respect to the laboratory performance for each batch of samples, as well as comparability of field and lab duplicates. Discussion will cover precision, accuracy, representativeness, comparability, and completeness as detailed in Worksheet #12 and detailed in this worksheet.

Precision

Laboratory precision is measured by the variability associated with duplicate (two) or replicate (more than two) analyses. One type of sample that can be used to assess laboratory precision is the LCS. Multiple LCS analyses over the duration of the project can be used to evaluate the overall laboratory precision for the project. In this case, the comparison is not between a sample and a duplicate sample analyzed in the same batch, but between LCSs analyzed in multiple batches.

Total precision is the measurement of the variability associated with the entire sampling and analytical process. It is determined by analysis of duplicate field samples and measures variability introduced by both the laboratory and field operations. FD samples and MSD samples will be analyzed to assess field and laboratory precision. For duplicate sample results, the precision is evaluated using the RPD. For replicate results, the precision is measured using the RSD. The formula for the calculation of RPD and PRD are provided as follows:

If calculated from duplicate measurements:

$$RPD = \frac{(C_1 - C_2)\ x\,100\%}{(C_1 + C_2)\ /\ 2} \qquad (1)$$

Where:

RPD = relative percent difference
C_1= larger of the two observed values
C_2 = smaller of the two observed values

If calculated from three or more replicates, use RSD rather than RPD:

$$RSD = (s\ /\ \bar{y})\ x\,100\% \qquad (2)$$

Where:

RSD = relative standard deviation
s = standard deviation
$\bar{y}$ = mean of replicate analyses

Standard deviation, s, is defined as follows:

$$S = \sqrt{\sum_{i=1}^{n} \frac{(yi - \bar{y})^2}{n-1}} \quad (3)$$

Where:

S = standard deviation
yi = measured value of the i^th replicate
$\bar{y}$ = mean of replicate analyses
n = number of replicates

Accuracy

Accuracy reflects the total error associated with a measurement. A measurement is considered accurate when the reported value agrees with the true value or known concentration of the spike or standard within acceptable limits. Analytical accuracy is measured by comparing the percent recovery of analytes spiked into an LCS to a control limit. For many methods of organic compound analysis, surrogate compound recoveries are also used to assess accuracy and method performance for each sample analyzed.

Both accuracy and precision are calculated for each analytical batch, and the associated sample results are interpreted by considering these specific measurements. The formula for calculation of accuracy is included in below as percent recovery (%R) from pure and sample matrices.

For measurements where matrix spikes are used:

$$\%R = 100\%\ x \left[\frac{S - U}{C_{sa}}\right] \quad (4)$$

Where:

%R = percent recovery
S = measured concentration in spiked aliquot
U = measured concentration in unspiked aliquot
Csa = actual concentration of spike added

For situations where a standard reference material (SRM) is used instead of or in addition to matrix spikes:

$$\%R = 100\% \ x \left[\frac{C_m}{C_{sm}} \right] \quad (5)$$

Where:

%R = percent recovery
Cm = measured concentration of SRM
Csm = actual concentration of SRM

Representativeness

Representativeness is a qualitative term that refers to the degree in which data accurately and precisely depicts the characteristics of a population, whether referring to the distribution of contaminant within a sample, a sample within a matrix, or the distribution of a contaminant at a site. Representativeness is determined by appropriate program design, with consideration of elements such as proper well locations, drilling and installation procedures, operations process locations, and sampling locations. Objectives for representativeness are defined for each sampling and analysis task and are a function of the investigative objectives. Assessment of representativeness will be achieved through use of the standard field, sampling, and analytical procedures.

Comparability

Comparability is a qualitative indicator of the confidence with which one data set can be compared to another data set. The objective for this QA/QC program is to produce data with the greatest possible degree of comparability. The number of matrices that are sampled and the range of field conditions encountered are considered in determining comparability. Comparability is achieved by using standard methods for sampling and analysis, reporting data in standard units, normalizing results to standard conditions, and using standard and comprehensive reporting formats. Complete field documentation using standardized data collection forms will support the assessment of comparability. Historical comparability will be achieved through consistent use of methods and documentation procedures throughout the project. Assessment of comparability is primarily subjective, and results should be interpreted by experienced environmental professionals with a clear knowledge of the DQOs and project decisions.

Completeness

Completeness is a measure of the amount of valid data obtained compared with the amount that was expected to be obtained under correct, normal conditions. It is calculated for the aggregation of data for each analyte measured for any particular sampling event or other defined set of samples (for example, by site) as set out in the DQOs. Valid data are data which are usable in the context of the project goals. Completeness is calculated and reported for each method, matrix, and analyte combination. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set. For completeness requirements, valid results are all results not qualified with an R-flag after a usability assessment has been performed. Completeness should not be determined only on the basis of laboratory data qualifiers. The goal for completeness is 95 percent.

Defined as follows for all measurements:

$$\%C = 100\% \; x \left[\frac{V}{T} \right] \qquad (6)$$

Where:

%C = percent completeness
V = number of measurements judged valid
T = total number of measurements

References

CH2M HILL. 2005. Quality Assurance Project Plan (Revised), Operable Unit 1, Quanta Resources OU1, Edgewater, New Jersey. October.

CH2M HILL. 2006a. *Quality Assurance Project Plan (Revised), Operable Unit 1, Quanta Resources OU1, Edgewater, New Jersey.* November.

CH2M HILL. 2006b. *Remedial Investigation/Feasibility Study Work Plan Addendum OU1, Quanta Resources Site, Edgewater, New Jersey*. July.

CH2M HILL. 2008a. *Quality Assurance Project Plan (Revised), Operable Unit 1, Quanta Resources OU1, Edgewater, New Jersey.* November.

CH2M HILL. 2008b. *Final Remedial Investigation Report, Quanta Resources Superfund Site, OU1*. August.

CH2M HILL. 2010b. *Technical Impracticability Evaluation, Quanta Resources Superfund Site, Operable Unit 1*. March.

CH2M HILL. 2013. *Quanta Resources Corporation Superfund Site, Operable Unit 1 (OU1) Vapor Intrusion—Results of the 2013 Monitoring Events at 115 River Road, 163 Old River Road, and 103 River Road.* May 21.

New Jersey Department of Environmental Protection (NJDEP). 2013. *Vapor Intrusion Technical Guidance and the associated NJDEP Vapor Intrusion Screening Level Tables*. March.

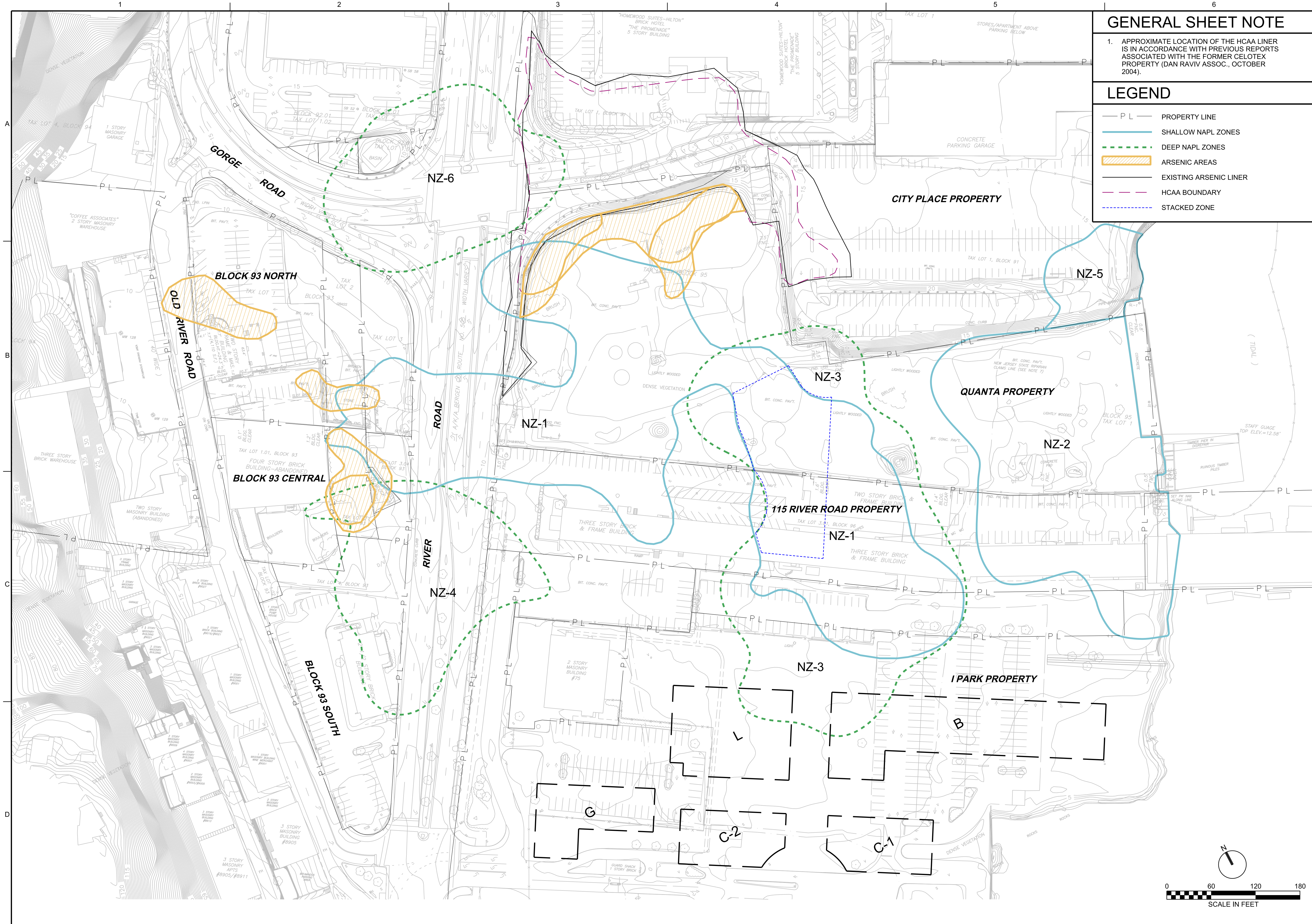
U.S. Environmental Protection Agency (USEPA). 2002. *OSWER Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils*.

U.S. Environmental Protection Agency (USEPA). 2008. *National Functional Guidelines for Organic Data Review*. June.

U.S. Environmental Protection Agency (USEPA). 2011. *Record of Decision, Operable Unit 1, Quanta Resources OU1, Edgewater, New Jersey.* October.

U.S. Environmental Protection Agency (USEPA). 2013. Office of Superfund Remediation and Technology Innovation (OSRTI), Vapor Intrusion Screening Level Calculator Tool. Version 3.0. May.

Figure 1
OU1 Base Map



VERIFY SCALE

BAR IS ONE INCH ON ORIGINAL DRAWING.

DATE	
PROJ	428872
DWG	FIGURE 1
SHEET	of

HONEYWELL INTERNATIONAL INC.
QUANTA RESOURCES CORPORATION
SUPERFUND SITE
Edgewater, New Jersey

FIGURE 1
OU1 BASE MAP

CH2MHILL

DSGN KD BLOCK
DR PA KARABAN
CHK
APVD

NO.	DATE	REVISION	BY	APVD

FILENAME: 10-C-R201-07-17-13.dgn PLOT DATE: 2013\07\31 PLOT TIME: 2:57:05 PM

Attachment 1
Standard Operating Procedures

ALS Environmental

DOCUMENT TITLE:	DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)
REFERENCED METHOD:	EPA TO-15
SOP ID:	VOA-TO15
REV. NUMBER:	20.0
EFFECTIVE DATE:	01/07/2013

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

EPA TO-15

SOPID:	VOA-TO15	Rev. Number:	20.0	Effective Date:	01/07/2013

Approved By: Wida Ang — Date: 12/14/12
Team Leader (VOA GC/MS) - Wida Ang

Approved By: Chris Parnell — Date: 12/14/12
Technical Manager (VOA GC/MS) - Chris Parnell

Approved By: Chaney Humphrey — Date: 12/14/12
QA Manager - Chaney Humphrey

Approved By: Kelly Horiuchi — Date: 12/17/12
Laboratory Director - Kelly Horiuchi

Archival Date: ______ Doc Control ID#: Non-Controlled Editor: ______

TABLE OF CONTENTS

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1) Scope and Applicability

1.1 This procedure is based on and incorporates the requirements detailed in EPA Compendium Methods TO-15 and TO-14A and is used to quantify a wide range of volatile organic compounds (VOCs) in gaseous matrices collected in gas collection bags (method modification) and specially prepared stainless steel canisters. This method typically applies to ambient concentrations of VOCs 0.50ug/m^3 (down to 0.10ug/m3 for low level ambient analyses) and above for the SCAN mode and 0.010ug/m^3 and above for the SIM mode; however, refer to Tables 3 and 3A for the specific laboratory initial calibration ranges for each target compound. The method requires VOC enrichment by concentrating up to one liter of a sample volume, with a virtually unlimited upper concentration range using dilutions from source level samples.

In this document, Tables 2 and 2A (see Note 1 below) list compounds that can be determined by this procedure along with their corresponding laboratory method reporting limits (MRLs) and method detection limits (MDLs). The reported MRL may be adjusted higher; however, the capability of achieving lower MRLs for specific project requirements must be thoroughly demonstrated (by an acceptable initial calibration and method reporting limit check standard) and documented as long as the MRL is higher than the current method detection limit for each compound. Additional compounds may be analyzed according to this procedure as described in the referenced methods as long as the requirements of this document are adhered to; however, if a compound is not listed in the TO-15 method, refer to Note 1 below. The number of samples that may be analyzed in a 24-hour period is about twenty. The number of sample results that may be reduced in an eight-hour day is approximately twenty.

<u>Note 1</u>: A number of compounds selected as target analytes are not included in the TO-15 Method; therefore the Florida Department of Health (FL DOH) required the laboratory to reference this Standard Operating Procedure (SOP) when reporting results for these analytes. However, the FL DOH disapproved of the SOP Code for this purpose, so CASS TO-15/GC-MS shall be referenced. Consequently, VOA-TO15 is otherwise known as CASS TO-15/GC-MS and shall be referenced, where necessary (on quantitation reports).

2) Summary of Procedure

2.1 The analytical method involves using a high-resolution gas chromatograph (GC) coupled to a mass spectrometer (MS). The GC/MS utilizes a linear quadrupole system, which allows for it to be operated by either continuously scanning a wide range of mass to charge ratios (SCAN mode) or by Select Ion Monitoring mode (SIM), which consists of monitoring a small number of ions from a specified compound list.

An aliquot of an air sample is concentrated on a solid adsorbent trap (either cryogenically or fan cooled glass beads or stronger adsorbents at higher temperatures) to collect the analytes of interest. To remove co-collected water vapor, the concentrated sample then goes through a water removal (dry purge) step. After the sample is pre-concentrated on a trap, the trap is heated and the VOCs are thermally desorbed onto a refocusing cold trap. The VOCs are then thermally desorbed onto the head of a capillary column once the cold trap is heated. The oven temperature (programmed) increases and the VOCs elute and are detected by the mass spectrometer.

Mass spectra for individual peaks in the total ion chromatogram are examined with respect to the fragmentation pattern of ions corresponding to various VOCs including the intensity of primary and secondary ions. The fragmentation pattern is compared with stored spectra taken under similar conditions, in order to identify the compound. For any given compound, the intensity of the primary fragment is compared with the system response to the primary fragment for known amounts of the compound. This method utilizes the internal standard calibration technique; refer to Section 3.16 for a complete definition.

3) Definitions

3.1 Cryogen A refrigerant used to obtain sub-ambient temperatures in the VOC concentrator and/or on front of the analytical column. Liquid nitrogen (cryogen) is used for this purpose and it has a boiling point of -195.8°C.

3.2 Gauge Pressure Pressure measure with reference to the surrounding atmospheric (barometric) pressure, usually expressed in units of psig. Zero gauge pressure is equal to atmospheric pressure.

3.3 MS-SCAN Mass spectrometric mode of operation in which the gas chromatograph (GC) is coupled to a mass spectrometer (MS) programmed to SCAN all ions repeatedly over a specified mass range.

3.4 MS-SIM Mass spectrometric mode of operation in which the GC is coupled to a MS that is programmed to scan a selected number of ions repeatedly [i.e., selected ion monitoring (SIM) mode].

3.5 Analytical Sequence The analytical sequence describes exactly how the field and QC samples in an analytical batch are to be analyzed.

3.6 Neat Stock Standard A purchased, single component assayed reference material having a stated purity used to prepare working calibration standards.

3.7 Stock Standards Solution A concentrated solution of one or more target analytes at a known concentration purchased from a reputable commercial vendor. Stock standard solutions are used to prepare working calibration standards.

3.8 Intermediate Calibration Standard A solution of one or more target analytes at a known concentration prepared either from one or more neat stock standards or from one or more stock standards solutions.

3.9 Working Calibration Standard A solution of all the target analytes at a known concentration prepared either from one or more intermediate calibration standards and/or from one or more stock standard solutions.

3.10 Calibration or Standard Curve A calibration or standard curve is a graph which plots the concentration of a compound (or an analyte) versus the instrument response to the compound.

3.11 Initial Calibration Verification (ICV) Standard A solution prepared in the laboratory containing known concentration(s) of analytes of interest. The solution is prepared from neat stock standards and/or stock standards solutions which are from a different source than the standards used to prepare the working calibration standards.

3.12 Continuing Calibration Verification (CCV) Standard A working calibration standard which is analyzed at specific intervals in order to verify that the instrument continues to meet the calibration criteria.

3.13 Field Sample A sample collected and delivered to the laboratory for analysis.

3.14 Manual Integration This term applies to a data file in which setpoints have been changed and reintegration has occurred under the changed setpoints; baselines have been adjusted; peak integration start and stop “ticks” have been changed; peak area, or peak height, are changed after the time of data collection and data file generation.

3.15 Batch Quality Control (QC) Batch QC refers to the QC samples that are analyzed in an analytical batch of field samples and includes the Method Blank (MB), Laboratory Control Sample (LCS) and Laboratory Duplicate (LD).

3.16 Internal Standard Calibration Compares the instrument responses from the target compound in the sample to the responses of specific standards (called internal standards), which are added to the sample or sample preparation prior to analysis. The ratio of the peak area (or height) of the target compound in the sample or sample preparation is compared to a similar ratio derived for each calibration standard.

3.17 May This action, activity, or procedural step is neither required nor prohibited.

3.18 Must This action, activity, or procedural step is required.

3.19 Shall This action, activity, or procedural step is required.

3.20 Should This action, activity, or procedural step is suggested, but not required.

3.21 SOP Standard Operating Procedure

3.22 Service Request A form generated, at the time of sample receipt, which details pertinent information such as client name, address, contact, client and laboratory sample identifications, sampling and receipt dates and times, requested analyses, sample type, canister pressures (initial and final), and the service request number (unique number for each submitted job) and serves as an inter-laboratory “custody” form which accompanies all samples throughout the laboratory.

3.23 Selectivity Selectivity of a method refers to the extent to which it can determine particular analyte(s) in a complex mixture without interference from other components in a mixture. Another definition is the extent to which a particular method can be used to determine analytes under given conditions in the presence of other components of similar behavior.

3.24 Limit of Detection (LOD) The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%. (DoD Clarification). For consistency purposes, the LOD may be referred to as the MDL once it is reported; however, full verification will be on file in the laboratory per the procedures detailed in this document.

3.25 Limit of Quantitation (LOQ) The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard. (DoD Clarification). For consistency purposes and since the LOQ and MRL are equivalent with regards to laboratory procedure, the LOQ will be referred to as the MRL in this document and once it is reported. Full verification will be on file in the laboratory per the procedures detailed in the document.

3.26 Detection Limit (DL) / Method Detection Limit (MDL) The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type 1 error) is 1%. (DoD Clarification). For consistency purposes, the DL may be referred to as MDL. Also, as far as reporting is concerned the MDL will be raised up (where necessary) to the verified LOD per the procedures defined in this document and reported accordingly.

4) Responsibilities

4.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP may perform analysis, interpretation and peer review of the results. Data reduction and/or peer review may be performed by another qualified employee. This employee must be familiar with the analytical technique and have completed a data review training plan to ensure familiarity with specific analysis and requirements.

The supervisor/manager must also ensure that method proficiency is documented initially and whenever significant changes in the instrument type, personnel, and matrix or test method are made. The department supervisor/manager or designee shall perform final review and sign-off of the data.

5) Interferences

5.1 Summa Canisters

Canisters shall be stored in a contaminant free location and shall be capped tightly during shipment to prevent leakage and minimize any compromise of the sample. The pressure/vacuum is checked prior to shipment and upon receipt from the field. Any problems with the sample from the field are noted and the Project Manager contacted.

Also, canisters must be cleaned and certified to be free from target analytes before being shipped to the field for sample collection. The procedure is described in detail in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters* (refer to this procedure as well as Section 12.5 for the acceptance criteria).

Current laboratory practice entails the segregation of 6L canisters into ambient (low) level and source levels. All the ambient canisters are used for low level (indoor air, ambient air) projects and not intentionally for soil gas, SVE monitoring, or other higher level applications. It may be necessary to "retire" an ambient canister and re-assign for source level use if high concentrations are encountered. This decision will be made by management based on analytical concentrations and what compounds were encountered at these levels. If the level of any analyte is detected above 5,000ug/m*3* in the ambient can, then the supervisor/team leader must be contacted to determine if the canister(s) is to be retired. If retirement is decided upon, make a notation on the sample tag (or other color coded tag) of each canister in question. The notation must contain the analyte, threshold levels and retirement from ambient use (initial and date notation) so that the canister conditioning/management department may properly execute the retirement.

5.2 Analytical System

The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running humidified zero air blanks. The use of non-chromatographic grade stainless steel tubing, non-PTFE thread sealants, or flow controllers with buna-N rubber components must be avoided.

5.3 Carbon Dioxide

Excessive levels of carbon dioxide present in a sample may interfere with analysis by freezing up the cryogenic trap. A smaller aliquot must be analyzed to eliminate this problem, or the sample should be analyzed using the higher temperature multi-adsorbent trapping technique which allows carbon dioxide to pass.

5.4 Gas Collection Bags

This procedure covers the use of gas collection vessels such as Tedlar® or Mylar® bags. However, due to the nature of these types of bags it is not recommended that clients use this option for ambient air samples. Sample collection bags made out of ®Tedlar have contaminants that are inherent to the manufacturing process. The two main contaminants are phenol and N,N-Dimethylacetamide. However, this only becomes a problem when the concentration levels in the sample are low ppbv such as ambient air monitoring samples where more of the sample usually has to be concentrated and analyzed. To minimize the loss of sample integrity, a 72-hour hold time has been incorporated into the procedure.

5.5 Glassware

Interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware results in discrete artifacts and/or elevated baselines in the detector profiles should be minimized. All glassware associated with this method must be scrupulously cleaned to avoid possible contamination. The cleaning shall be performed in accordance with the procedure outlined in the *SOP for Glassware Cleaning.* The use of high purity water, reagents, and solvents helps to minimize these problems.

6) Safety

6.1 Each compound, mixture of compounds, standards, and surrogates, as well as samples, should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest level possible through the use of gloves (to minimize absorption through the skin) and hoods (to minimize inhalation). Refer to the laboratory's Environmental, Health and Safety Manual as it makes reference to the safe handling of chemicals, MSDS location, and the laboratory waste management plan for the safe disposal of chemicals and samples.

6.2 Material Safety Data Sheets (MSDS)

The analyst should consult MSDS for compounds being handled in the course of this procedure, and be familiar with proper safety precautions to be followed when handling hazardous chemicals. Care should be taken when handling standard material in a neat or highly concentrated form.

6.3 Liquid Nitrogen

Liquid nitrogen can cause serious tissue damage (frostbite) with only a few seconds of contact. The valves on the cryogen dewars should be opened slowly so leaky fittings can be identified. Neoprene or leather gloves should be worn when turning valves and handling tubing and fittings that have been in contact with the cryogen.

6.4 Protective Clothing

Personal protective clothing (safety glasses, gloves and lab coat) are required when preparing standards and handling standard material in neat form.

6.5 Pressurized Gases

The use of pressurized gases is required for this procedure. Care should be taken when moving cylinders. All gas cylinders must be secured to a wall or an immovable counter with a chain or a cylinder clamp when not in use. The regulator should never remain on small "D" size cylinders following use. Sources of flammable gases (i.e. pressurized hydrogen) should be clearly labeled.

6.6 Syringes

The proper use of syringes should be part of employee training for this SOP. Care should be taken to avoid personal injury as a result or improper handling techniques.

7) Sample Collection, Containers, Preservation, and Storage

7.1 Air samples are collected in the field and delivered to the laboratory and shall be collected in either a specially prepared, leak-free, stainless steel pressure vessel (with valve) of desired volume (e.g., 6L) or a sample collection bag (Tedlar). Canister samples may either be grab or time integrated (using a variable flow controller, refer to the *SOP* *for Flow Controllers and Critical Orifices)* utilizing the canister vacuum to draw the sample. Bags require the use of an upstream pump or a "lung machine."

7.2 There are no special preservation requirements for either canisters or bags. However, bags should be stored in an environment free from puncture or deterioration sources (by hanging them from clips), labeled with the specific service request number, in accordance with the *SOP for Laboratory Storage, Analysis and Tracking*. Canisters should be stored on the appropriate shelves until they are to be analyzed.

7.3 Sample collection bags must be analyzed within 72 hours from the confirmed time of sampling. Samples received by the laboratory shall be analyzed within 30 days of sampling or sooner if project specific requirements dictate. Programs, which have shorter recommended or required hold times, include the Department of Toxic Substances Control (DTSC), which advises a 72 hour hold time. The Minnesota Pollutions Control Agency (MPCA) and EPA Region 9 both require a 14 days hold time. Additionally, the MPCA does not allow the use of Tedlar bags for sampling or sample dilution. The DTSC requirement is an advisory notice, but the laboratory shall make every effort to comply. However, the following statement shall be added to each report where sample analyses do not meet the 72 hour hold time and the client project is intended to comply with DTSC requirements. *"The recommended 72-hour hold time for the analysis of TO-15 was exceeded per the DTSC and LARWQCB Advisory – Active Soil Gas Investigations document dated January 28, 2003; however, this specific hold time statement is advisory and not considered as regulation. In addition, the samples were analyzed within the EPA Method TO-15 stated requirement of 30 days."*

8) Apparatus and Equipment

8.1 Additional instruments and/or differing models may be utilized as long as they are equivalent and meet the minimum requirements of this document.

8.2 Gas Chromatograph (GC)

An instrument capable of temperature programming, with a column oven that may be cooled to sub-ambient temperature at the start of the gas chromatographic run to result in the resolution of the VOCs.

Hewlett Packard 5890 Series II Plus
Hewlett Packard 6890 Series
Hewlett Packard 6890A Series
Agilent 6890N Series
Agilent 7890A Series

8.3 Autosampler

Tekmar-Dohrmann AUTOCan Autosampler:	14-ACAN-074
Concentrating Trap (cryogenic trap, built-in):	14-6938-020
Cryofocusing Module w/split valve:	14-6520-A00
GAST Vacuum Pump:	DOA-P104-AA or equivalent

8.4 Mass Spectrometer (MS)

A MS capable of scanning from 34 to 350 amu every second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for Bromofluorobenzene (BFB) which meets all of the criteria when 50ng or less of BFB is injected onto the GC/MS system.

Hewlett Packard 5972 Series
Hewlett Packard 5973 Series
Agilent 5973N
Agilent 5973 *inert*
Agilent 5975B *inert*
Agilent 5975C *inert*

8.4.1 Ionization Gauge Controller

- Agilent: 59864B
- Granville-Phillips 330 Ionization Gauge Controller: 330001/2/3
- Hewlett Packard Ionization Gauge Controller: 59864B

8.5 Analytical Column

Any analytical column capable of separating the compounds of interest may be used. The capillary column should be directly coupled to the source of the mass spectrometer. The following are suggested columns; an alternative column may be used as long as sufficient peak resolution and separation is achieved.

- Restek Rxi-1ms Fused Silica Capillary Column; 30m x 0.25mm ID 1.0µm film thickness

OR

- Restek Rxi-1ms Fused Silica Capillary Column; 60m x 0.25mm ID 1.0µm film thickness

8.6 Data Systems

IBM-compatible PC with Windows 95/98/NT/XP/7 (Microsoft Office EXCEL version 2003 or newer) and Hewlett Packard Chemstation software including EnviroQuant with Extracted Ion Current Profile (EICP), National Institute of Standards and Technology (NIST) library (2002 version or newer) or equivalent.

8.7 Canister Pressurization Station

Vacuum/Pressure Gauge [0 to –30 inHg; 0-90 or 100 psig]

8.8 Canister Sampling Devices

Refer to the *SOP for Flow Controllers and Critical Orifices* for specific calibration and other pertinent information.

- VICI Condyne Model 300 Flow Controller

- Critical Orifices (Laboratory manufactured)

8.9 Gas Collection Devices

- Lab Commerce, Aerosphere Model S6L, 6.0L Summa Passivated Canisters or equivalent
- Lab Commerce, Stabilizer Model 22.4L, 2.4L Canisters or equivalent
- Restek Corporation, #24203, 3.0L Silco Canisters or equivalent
- Tedlar bags – 0.5L, 1L, 3L, 5L, 10L, 25L, and 40L (other sizes are available; however, the volumes that are listed encompass the majority of the bags supplied and the samples submitted to the laboratory).

8.10 Dynamic Dilution System

- Entech Dynamic Diluter Model 4620A
- Toshiba laptop computer Model 2210CDT/6.0 and Software NT460

9) Standards, Reagents, and Consumable Materials

9.1 Reagents and Equipment

9.1.1 UHP Grade Helium (99.999%) (GC carrier gas, preconcentrator purge/sweep gas, pressurization gas)
9.1.2 Cryogen - Liquid nitrogen from bulk tank or 50 psig dewars (used to cool preconcentrator traps)
9.1.3 UHP/Zero Grade Air (canister pressurization)
9.1.4 ASTM Type II Water, DI water or equivalent
9.1.5 UHP Grade Nitrogen (99.999%) (additional pressurization gas, based on other methods requested – modification to method)

9.2 Standards

Standards are prepared for both SCAN and Selective Ion Monitoring (SIM) modes according to the procedures detailed in this section. The preparation of standards for the analysis of air samples is carried out by following the procedure, "Preparation of Gas Phase Standards for Ambient Air Analysis", Application Note, Spring 96, Vol. 6.5, *Tekmar*-DOHRMANN AutoCan Users Manual. Neat standards that are used for making trace gas standards must be of high purity; generally a purity of 98 percent or better is commercially available.

9.2.1 Instrument Performance Check, Internal Standard and Surrogate Spiking Mixture
Prepare a standard solution of p-Bromofluorobenzene (BFB-used as both a tune check and surrogate compound), bromochloromethane, chlorobenzene-d5, and 1,4-difluorobenzene, 1,2-dichloroethane-d4(surrogate), and toluene-d8(surrogate) at 500μg/m^3 each in humidified zero air (Section 9.2.1.2). Prepare this standard according to the procedure outlined in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note. This standard may also be prepared from a neat cocktail as in Section 9.2.2.2.1 or as stated in Section 9.2.1.3.

9.2.1.1 An intermediate standard is prepared from neat compounds in a glass static dilution bottle (SDB). After the volume of the SDB is determined, calculate the mass of each compound to be spiked to achieve a final concentration of 5.0μg/mL. Then use the density of each neat compound to calculate the microliter amount to be spiked into the SDB. The SDB is then heated for a minimum of one hour at ~60°C to completely volatilize all components.

Concentration of the intermediate standard prepared in a SDB is 5.0µg/mL. The amount required to achieve this concentration is determined through the use of the following equation.

$$A = \frac{(C)(V)}{D} \qquad \text{(Equation 1)}$$

Where:

A	Amount of each compound required to achieve the desired concentration of the standard in the SDB (µL)
C	Desired concentration of SDB (µg/mL)
V	Actual volume of the SDB (mL)
D	Density of the compound in question (µg/µL)

Example:

Calculate the amount of neat bromochloromethane needed to achieve the final concentration of 5.0µg/mL of that compound in the SDB.

V = 2010mL
D = 1934.4µg/µL
C = 5.0µg/mL

$$A=\frac{\left(5.0\frac{\mu g}{mL}\right)2010mL}{1934.4\frac{\mu g}{\mu L}} = 5.2\mu L$$

Density (µg/µL)	Compound
1934.4	Bromochloromethane
1170.1	1,4-Difluorobenzene
1157	Chlorobenzene-d5
1307	1,2-Dichloroethane-d4
943	Toluene-d8
1593	BFB

9.2.1.2 The Working standard is prepared in a Summa canister by spiking an aliquot of the stock SDB standard (Section 9.2.1.1) using a heated gastight syringe. Connect a cleaned, evacuated Summa canister to a source of pure diluent gas (humidified zero air) using a Teflon line with a stainless steel tee directly above the canister valve. One port of the tee is fitted with a septum. Spike the SDB stock and following removal of syringe a small flow of diluent gas to flush the spike into the can.

Pressurize the can to positive 83.3 psig with humid zero air, and allow the contents to equilibrate for approximately 24 hours before using.

Concentration of the working standard prepared in a Summa canister is 500ng/L. The final pressure of the canister is 83.3psig; therefore, the pressurized volume is 40L, which is obtained through the use of the following equation.

PV = PDF(V) (Equation 2)

Where:

PV Pressurized canister volume (L)

PDF Pressure Dilution Factor, where PF = $\frac{P_{atm} + P_f}{P_{atm} + P_i}$

P_f Final Canister Pressure

P_i Initial Canister Pressure

V Volume of canister at 1atm

P_{atm} Atmospheric Pressure = 14.7psig

Example:

$$\frac{14.7 + 83.3}{14.7 + 0}(6L) = 40\text{L}$$

In order to prepare the canister with a concentration of 500ng/L, it must be determined how much of the intermediate standard is required. This is achieved through the use of the following equation.

$$A = \frac{(F)(V)}{(C)\left(1000\frac{ng}{\mu g}\right)} \qquad \text{(Equation 3)}$$

Where:

F Desired concentration of working standard (ng/L)
V Pressurized Volume of Canister (L)
C Concentration of prepared SDB (µg/mL)
A Amount of standard (mL) of the SDB required to obtain the desired working standard concentration

Example:

$$A = \frac{500\frac{ng}{L}(40L)}{\left(5.0\frac{\mu g}{mL}\right)\left(1000\frac{ng}{\mu g}\right)} = 4mL$$

9.2.1.3 Currently the working standard is purchased in a cylinder at a certified concentration of 500ng/L (prepared by Spectra Gases Inc, Alpha, NJ).

9.2.1.3.1 For SCAN analyses, the working standard is filled directly into a summa canister to a pressure of 70 to 80 psig.

9.2.1.3.2 For SIM analyses, the working standard is diluted and pressurized with humid zero air to the desired concentration using Equation 2 in Section 8.2.1.2. Typical concentrations will be 20ng/L, 40ng/L or 50ng/L.

9.2.2 Initial Calibration (ICAL) Standard Prepare the primary source calibration standards in Summa canisters with nominal concentrations of 4ng/L (optional), 20ng/L and 200ng/L for analyses in SCAN mode and 0.1ng/L and 5.0ng/L, 20ng/L for analyses in Selective Ion Monitoring (SIM) mode for each of the target analytes. Differing injection volumes will create the standard concentrations listed in Tables 3 (SCAN) and 3A (SIM) of this document. The full list of analytes which are analyzed according to this method can also be found in Tables 2 (SCAN) and 2A (SIM).

Standards are prepared by diluting the stock standard with humid zero air into a Summa canister. The stock standard is a certified custom-blended cylinder (prepared by Spectra Gases Inc, Alpha, NJ). Refer to Tables 3 and 3A for the list of analytes and certified concentrations in the purchased cylinder.

9.2.2.1 Working standards are prepared into Summa canisters using the Entech Dynamic Diluter. Turn on the power to the diluter one hour prior to using to allow for the components to come to thermal equilibrium. Connect the computer and start the software. Connect a Zero Air source to the humidification chamber (flow controller #1). Connect stock standard cylinder#1 to flow controller #2 inlet. Open the cylinder valves. Adjust the inlet pressures to 50 to 60psig.

Standard Concentration Selection: The concentration of the two working standards prepared in Summa canisters should be 200ng/L and 20ng/L or 200ng/L, 20ng/L and 4ng/L (depending on the dynamic range of the initial calibration-include 4ng/L if a 0.1ng on column standard is desired or this standard may be used for the 0.5ng/L concentration as well) for SCAN and 0.1ng/L and 5.0ng/L, 20ng/L for SIM.

Position 1 – Total Air Flow (Zero Air)
Position 2 – Standard Flow (Purchased Standard One)
Position 3 – Standard Flow (Purchased Standard Two if Applicable)
Position 4 – Total Air Flow (Zero Air) (utilized if preparing a two dilution standard)

Position 5 – Diluted Standard Flow (utilized if preparing a two dilution standard)

Step1: Determine the required flow rate of the stock standards (positions #2 and #3). The range must be from 5 to 50sccm (standard cubic centimeters per minute, same as ml/min). The flows listed below are guidelines to be used for the default standard flow (based on the desired standard concentration) and were chosen based on the ultimate final dilution required and limitations of the Dynamic Diluter (flows must be from 150 to 2000ml/min.).

Desired Standard Conc.	Default Standard Flow
200ng/L	50ml/min
100ng/L	50ml/min
20ng/L	20ml/min
5.0ng/L	10ml/min
4.0ng/L	8ml/min
0.1ng/L	10ml/min (See Note 1 below)

Note 1: For the 0.1ng/L standard (or another two dilution standard), a slightly different procedure is performed. In order to prepare this standard, two dilutions must be performed. Unscrew the cover of the dilutor and connect the first mass flow controller as well as the tubing to re-route the first dilution output from the final standard Summa canister to the 2nd dilution chamber. Refer to example 2 for the calculation guidelines to prepare a two dilution standard.

Example 1: Prepare a 200ng/L working standard. The concentration of each stock standard is 1000ng/L.

Step 2: Determine the required dilution factor for each stock.
Dilution factor = Stock Conc. (ng/L) / Desired Standard Conc. (ng/L)
Dilution Factor = 1000ng/L / 200ng/L = 5

Step 3: Calculate Total Flow
Total Flow= (stock std. flow-see table above)*(Dilution Factor)
Total Flow=50ml/min*5 = 250ml/min

Step 4: Calculate Diluent Air Flow
Air Flow=Total Flow-(Sum of stock std. flows-purchased cylinders)
Air Flow=250ml/min-(50+50)ml/min = 150ml/min

Example 2: Prepare a 0.1ng/L working standard. The concentration of each stock standard is 1000ng/L.

Step 2: Determine the required total dilution factor for the 0.1ng/L standard.
Dilution factor = Stock Conc. (ng/L) / Desired Standard Conc. (ng/L)
Dilution Factor = 1000ng/L / 0.1ng/L = 10,000

The two dilutions must be performed which total the dilution factor calculated above. Since the flow for the Diluter is restricted to a maximum of 2000ml/min, the total flow (as calculated in Step 3 below) cannot exceed 2000ml/min; therefore, the dilutions must be chosen accordingly.

Step 3: Calculate Total Flow
Total Flow = (stock std. flow-see table above)*(Dilution Factor)
Total Flow (Dilution 1) = 10ml/min*200 = 2000ml/min

For the 2nd dilution take the stock standard flow selected for dilution 1 for the two purchased cylinders (10ml/min each based on the desired final concentration) and add them together (10ml/min + 10ml/min for 20ml/min) to get the stock standard flow for the 2nd dilution.

2nd Dilution Factor Needed = Total Dilution/1st Dilution
2nd Dilution Factor = 10000/200(1st dilution) = 50
Total Flow (Dilution 2) = 20ml/min*50 = 1000ml/min

Step 4: Calculate Diluent Air Flow
Air Flow=Total Flow-(Sum of stock std. flows-purchased cylinders)
Air Flow=2000ml/min-(10+10)ml/min = 1980ml/min (Dilution 1)
Air Flow=1000ml/min-20ml/min = 980ml/min (Dilution 2)

Position 1 = 1980ml/min
Position 2 = 10ml/min
Position 3 = 10ml/min
Position 4 = 980ml/min
Position 5 = 20ml/min

Step 5: Enter flow rates in the appropriate fields in the Entech software. Start flows by clicking the "GO" button in the top right of the window. Allow flows to equilibrate for at least fifteen minutes, then attach an empty canister to the outlet port and open the valve. The outlet pressure will be displayed in the lower right of the window, in units of psia. Close the canister valve when the pressure reaches 30psia. There is a relief valve on the diluter that will open when the pressure reaches 35psia, so the canister will still be usable if the valve is not closed in time.

9.2.2.2 When analysis of additional (extra) compounds are requested which are not in the purchased stock cylinders, the following preparation instructions should be used. In addition, the internal standard / surrogate standard may also be prepared in this manner (Sections 9.2.2.2.1 – 9.2.2.2.2) as mentioned in Section 9.2.1.

9.2.2.2.1 *Equi-mass "soup"* (contains compounds in equal mass amounts) or *cocktail* prepared from the neat compounds for a large number of components. If additional SIM compounds are requested, the same cocktail may be used.

Cocktail Preparation:
Step 1: This cocktail is prepared by combining 25mg of each neat compound into a small glass vial. Use a microliter syringe to transfer each compound, cleaning with solvents in between.

Put the vial in the freezer between aliquots to minimize volatilization. Take the density of each compound into account to determine the actual amount of each compound to spike into the cocktail by using the following equation.

$$S = \frac{A}{D} \qquad \text{(Equation 4)}$$

Where:

S Actual spike amount (μL)
A Desired amount for each compound (mg)
D Density (mg/μL); refer to Table 2 for the density

Example: The actual volume of acrolein to add to the cocktail is calculated by the following.

$$S(\text{Acrolein}) = \frac{25mg}{\left(0.840\frac{mg}{\mu l}\right)} = 29.8\mu L$$

Step 2: The concentration of each compound in the cocktail is determined by the following equation.

$$C = \frac{A}{V}\left(1000\frac{\mu g}{mg}\right) \qquad \text{(Equation 5)}$$

Where:

C Concentration of cocktail (μg/μL)
A Amount of each compound (mg)
V Final volume of cocktail (total spike volumes of each compound) (μL)

Example:

$$C = \frac{25mg}{631.8\mu L}\left(1000\frac{\mu g}{mg}\right) = 39.569\mu g/\mu L$$

8.2.2.2.2 *An intermediate standard* is prepared from neat compounds by spiking individual compounds into a glass static dilution bottle (SDB) as described in Section 9.2.1.1 or spiking an aliquot of a cocktail into the SDB. The spike amount of a cocktail is determined by using the following equation.

$$S = \frac{C_1 V}{C_2} \qquad \text{(Equation 6)}$$

Where:

S Spike amount required in order to obtain the desired concentration (μL)
C_1 Desired concentration of SDB (μg/mL)
C_2 Concentration of cocktail (μg/μL)
V Volume of SDB (L)

Example: Determine the spike amount of the cocktail required to achieve the desired intermediate standard concentration.

$$S = \frac{\left(1\frac{\mu g}{ml}\right)(2010ml)}{27.81\frac{\mu g}{\mu L}} = 72.28\mu L$$

9.2.2.2.3 *Intermediate Standard Preparation (Gaseous Compounds)* As an alternative to the glass SDB method, if the extra compounds needed to be analyzed are gases at room temperature, use a gastight syringe to prepare an intermediate standard in a 1L Tedlar bag filled with humidified zero-grade air. Use the molecular weight of the compound to calculate the microliter amount to be spiked into the bag to achieve desired concentration. The spike amount is determined by using the following equation.

$$S = \frac{C * V * 24.46}{M * \left(1000\frac{ng}{\mu l}\right)}$$

S Spike amount required in order to obtain the desired concentration (μl)
C Desired concentration (ng/L)
V Volume of the Tedlar Bag (1L)
M Molecular Weight of the compound
24.46 Molar Volume of gas at 25°C, 1atm

Example:

Make a 100,000ng/L intermediate standard of Chloro-difluoromethane (Freon22) in a Tedlar Bag, where M=86

$$S = \frac{100{,}000\,\frac{ng}{L} * 1L * 24.46}{86 * \left(1000\,\frac{ng}{\mu l}\right)} = 28.44\mu l$$

9.2.2.2.4 *The Working standard* for extra compounds is prepared in a Summa canister by spiking an aliquot of the intermediate standard (glass SDB or Tedlar bag) using a heated gastight syringe. The preparation of these standards shall follow the instructions detailed in Section 9.2.1.2. The concentrations for working standards are usually 20 and 200ng/L, however different concentrations can be chosen which work best for a particular project.

9.2.3 Initial Calibration Verification (ICV) - (Laboratory Control Sample - LCS) Prepare a secondary source standard (either a different manufacturer or different lot from the same manufacturer as the initial calibration standard) using the same procedures as the primary source. The ICV/LCS working standard should contain each target analyte present in the calibration working standard. Prepare the ICV/LCS working standard at a concentration of 200ng/L. Differing injection volumes account for the allowed concentrations listed in Table 4 for SCAN and 4A for SIM. The preparation of this standard shall follow the instructions detailed in Section 9.2.2, using the certified second-source standard cylinder.

9.2.4 Continuing Calibration Verification (CCV) Standard The CCV is the same as the initial calibration working standards detailed in Section 9.2.2.

9.2.5 Screening Standards Recommended procedure: Prepare a 0.5ug/mL and/or a 3.0ug/mL concentration standard so that the GC may be calibrated utilizing a few levels (may include approximately 0.5ng, 150ng and 600ng). However, other concentrations can be prepared depending on the desired range.

Any of the desired standard concentrations (primary and secondary) may change as long as the equations and the appropriate densities remain the same.

9.3 Storage and Expiration Dates

All standards that are to be stored in a freezer shall be stored at ≤-10°C for DoD projects.

- Neat Stock Liquids are stored at < -10°C (-10°C to -20°C) as specified by the manufacturer or for a period of five years.
- Equi-Mass Primary Stock Standard is a cocktail or soup of neat compounds (containing compounds in equal mass amounts) used to in preparing intermediate gas phase standards and shall be stored in the freezer at < -10°C (-10°C to -20°C) for up to six months. This is assuming that the soup is sealed with a septum-containing screw cap or Mininert™ valve. The selection of the compounds for the soup should be performed in accordance with the guidelines in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note.

- Purchased Stock Standards Cylinders must be stored at laboratory temperature for a period of 2 years or as specified by the manufacturer before vendor re-certification or purchase of new standards.
- Intermediate Calibration Standards prepared by static dilution must be stored in an oven at a temperature of approximately 60°C to ensure analyte vaporization. Every time a standard is prepared from the static dilution bottle (SDB), the concentration changes. To increase the useful lifetime of an SDB standard, remove volumes of 25mL or less. The volume removed can be manipulated by increasing the SDB concentration or by adjusting the canister final volume/pressure. Depending upon the volume removed, an SDB intermediate standard is stable for approximately two months as long as new working standards made from this standard continue to meet acceptance criteria. These bottles must be in the oven for a minimum of one hour prior to use in preparing working standards. The guidelines for the storage and expiration date for the intermediate calibration standards are stated in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note.
- Prepared Stock / Intermediate Calibration Standards prepared in Summa canisters (1000ng/L) may be stored at laboratory conditions for up to three months in an atmosphere free of potential contaminants. Upon preparation, canister standards should be allowed to sit for approximately 24 hours prior to use in order for equilibration to take place. Shorter equilibration periods may be necessary and acceptable as long as performance criteria are met.
- Calibration or Working Calibration Standards prepared in canisters may be stored at laboratory conditions for one month in an atmosphere free of potential contaminants. Upon preparation, canister standards should be allowed to sit for approximately 24 hours prior to use in order for equilibration to take place. Shorter equilibration periods may be necessary and acceptable as long as performance criteria are met.

10) Preventive Maintenance

10.1 A maintenance log will be kept documenting maintenance performed on each analytical system. The serial numbers of each instrument shall be recorded, and each log entry must include a description of the maintenance performed and be initialed by the analyst performing or observing/authorizing maintenance by an outside contractor.

The instrument maintenance log must be kept current. An entry shall be made in the appropriate log every time maintenance is performed (no matter the extent). The entry in the log must include:

(a) The date of maintenance
(b) Who did the maintenance
(c) Description of the maintenance
(d) Proof that the maintenance activity was successful

A notation of a successful tune and continuing calibration or initial calibration and the file number that accompanies the data will serve as proof that the maintenance is complete and the instrument is in working order.

The extent of the maintenance is not important, however, it is important that a notation be included for each maintenance activity such as changing a column, tuning the instrument, changing the pump oil, cleaning the source, ordering a part. In addition, a notation should be made in the logbook stating that no samples were analyzed during the days that the instrument was down and no active maintenance was being conducted (i.e., where no other notation was made in the logbook for those days).

10.2 Concentrating Trap

Routine maintenance includes periodic solvent cleaning of the Silco steel lines in the valve oven if contamination is suspected. Also, periodic replacement of the multi-sorbent or partial replacement of the trap if analyte specific deterioration is detected is required. For specific trap information refer to the instrument maintenance logbook and electronic method manual.

After repacking, the trap should be baked at 265°C for a minimum of two hours (or until a clean blank is generated) and a partial repacking requires baking (at 265°C) the trap for a minimum of 20 minutes (or until a clean blank is generated).

10.3 GC System

Column performance is monitored by observing both peak shapes and column bleed. Over time, the column will exhibit a poor overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced (see Section 7.4). Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column.

Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column-cutting tool. When removing any major portion of the column, which will affect the retention times and elution characteristics, a change in instrument conditions may be required to facilitate nominal analytical activity.

Declining performance can also be due to ineffective column ferrules, which should be replaced when a tight seal around the column is no longer possible. This can be detected with the use of a leak detector.

10.4 Mass Spectrometer

The Mass Selective Detector (MSD) ion source requires periodic cleaning to maintain proper performance. Symptoms of a dirty ion source include difficulty keeping the MSD in tune and fluctuating internal standard areas. The vacuum system should be serviced every six months, including changing the pump oil and checking the molecular sieve in the back-streaming trap.

10.5 Instrument Tuning

The instrument is tuned with guidance from the procedure described in the HP Operations Manual, when necessary.

10.6 Computer Troubleshooting

Computer care and troubleshooting is conducted by the IT department. Refer to Section 8.5 for the computer hardware and software requirements.

Computers are selected to meet or exceed operating system and or acquisition software requirements. Periodic upgrades of memory are performed to maintain or improve system performance and reliability. Upgrades may be performed on systems until instrument hardware configurations become the limiting factor.

Basic Troubleshooting Outline:

1) Document occurrence and severity in IT Log
2) Interview user(s)
3) Investigate any available logs (Event Logs, Acquisition Logs, etc.)
4) Determine if problem is isolated (single user or acquisition) or widespread (multi user or network).
5) If multiple possibilities exist for cause, then eliminate in systematic manner.
6) Hardware issues are addressed with component replacement (beginning with most suspect portion).
7) Software issues are addressed first with internet investigation (user blogs, software source updates/findings).
8) Network issues are investigated from the Server, to Switch, to Network Card; utilizing all available managed devices to help discover possible failure points.
9) In some cases, system corruption may require reload or complete system replacement.
10) Finalize documentation in IT Log with actions taken
11) Perform periodic follow-up with User and review any log found to have suspect events that suggested source of issue.

11) Procedure

11.1 Sufficient raw data records must be retained on file of all laboratory analyses described in this document including passing QC canister checks, tune checks, instrument calibrations, verifications, sample analyses and dilutions, QC checks, and method detection limit studies. The information that is required includes: analysis/calibration date and time, test method, instrument, sample identification, analyte identification, analyst's initials, concentrations and responses, as well as standards used for the analysis and calibrations, all manual calculations including sample dilutions and manual integrations to permit reconstruction of analyses. Information entered and reported on the quantitation report and instrument run log must be complete and accurate. All data shall be obtained following defensible and ethical practices in accordance with the most recent Quality Assurance Manual and the *SOP for Laboratory Ethics and Data Integrity*.

Note: All data records must explicitly connect data to the initial instrument calibration. This includes all samples, continuing calibrations and QC samples.

11.2 Sample Preparation

The pressure/vacuum is checked and the canister pressurized upon receipt by the laboratory, as needed. When necessary, canisters shall be pressurized with humidified zero grade air. However, if the samples are to be analyzed in accordance with EPA Method 3C then the samples must be pressurized with UHP Helium (refer to Section 11.12 for additional information). The client must be made aware of this in advance and given the option of either submitting two canisters for analysis or receiving a report with qualified results (TO-15 Modified).

Depending on the size of the canister and location of sampling and as specified in the SOP below, samples may be pressurized to approximately 1.0psig to 3.5psig. Additional information may be found in the *SOP for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters*. Initial and final pressures are recorded in LIMS and should be repeated on the back of the sample tag. The dilution factor created by filling the sample canister is calculated using equation number 12 in Section 13.8.

11.3 Screening

The analyst must screen a sample or subset of samples if the source is of unknown origin. Typically, if the source is known to be indoor or ambient outdoor air, no screening is necessary. However, if screening is required make sure that the instrument is calibrated. A single point calibration is sufficient; however, the instrument may be calibrated utilizing a two point calibration. The ICAL points are recommended to be at approximately 0.5ng, 150ng and/or 600ng spanning the desired dynamic range. Refer to Section 9.2.5 for additional information.

Inject a 1mL or smaller aliquot of each sample into a GC/flame ionization detector (FID) system that has been calibrated with a standard containing a subset of the target analytes. This subset represents the most commonly found compounds in air samples, such as acetone, trichloroethylene, and toluene. Use the results to determine the maximum volume of sample to be analyzed by TO-15 by utilizing the following equation. Dilutions may be prepared as necessary according to Section 11.12.1.

$$I = \frac{C}{H}$$ Where:

I Injection volume (mL)
C Maximum calibration level (ng on column)
H Compound screening concentration (ng/mL)

Example: Select the compound with the highest concentration (toluene = 1.0ng/mL). If the upper calibration level is 100ng on column, then the following calculation determines the maximum injection volume to analyze.

$$\frac{100ng}{1.0ng/mL} = 100\text{mL maximum injection volume}$$

11.4 Analytical Sequence and Data System Setup

11.4.1 Data System For the Tekmar AUTOCAN, fill in the sequence log of the Teklink program with the appropriate information. Refer to the Section 11.5.1 for the operating parameters.

For HP Chemstation, load the appropriate acquisition method for the GC/MS in the top window of the Chemstation program. Suggested GC/MS operating parameters are given in Section 11.5.2.

11.4.2 Analytical Sequence The analytical sequence must be completed for the analysis of ≤20 (19 samples including dilutions with one laboratory duplicate) field samples. A method blank (MB) shall be run to monitor for laboratory introduced contamination. There must be at a minimum a laboratory duplicate (LD) analyzed in each batch to access batch precision. The following generalized analytical sequence is to be followed:

Analytical Sequence Guideline

With Calibration	Tune Check[1] Calibration Standards (5 Standards Minimum) ICV Standard[2] (Acts as the ICV and LCS) QC Canister Checks[6] MB[7] Sample(s) – 1-19 Laboratory Duplicate[4]
With Continuing	Tune Check[1] CCV Standard[5] QC Canister Checks[6] MB[7] LCS[3] MRL Check Standard[8] Sample(s) – 1-19 Laboratory Duplicate[4]

[1] The instrument performance check solution must be analyzed initially and once per 24 hour (or as specified by the project) time period (sequence / tune window) of operation. All analyses for a sequence must be initiated (injected) prior to the expiration of the tune window.

[2] In this scenario, the ICV may also be evaluated as the LCS (differing acceptance criteria).

[3] An LCS shall be analyzed at a rate of 1 in 20 or fewer samples. The LCS is the second source calibration check standard analyzed at the lower end of the calibration curve (below the midpoint).

[4] A laboratory duplicate must be analyzed at a rate of 1 per 20 or fewer samples. The duplicate must be rotated among clients, whenever possible. Also, a duplicate laboratory control sample may be analyzed to assess precision to meet project requirements or due to sample matrix effects.

[5] A CCV must be analyzed at the beginning of every analytical sequence.

[6] Any number of QC check canisters may be analyzed in the sequence to determine a canister cleaning batch or batches acceptability.

[7] Any of the QC Check Canisters may serve as the method blank as long as the minimum requirements detailed in this document are met. A method blank shall be analyzed at a rate of 1 in 20 or fewer samples.

[8] A MRL check standard may be analyzed with each batch of 20 or fewer samples (when an initial calibration is not analyzed within the same batch). Additional information is included in Section 11.18.

Note: Client project batch specifications may require certain modifications to the analytical sequence; however, a batch may not be more lenient than that which is specified in this document.

11.5 Conditions

11.5.1 Sample Collection Conditions The suggested settings and system parameters are as follows:

Adsorbent Trap

Set Point:	35˚
Sample Volume:	up to 1L
Dry Purge:	300mL
Sampling Rate:	100mL/min (utilize for a sample injection volume of >100mL); 40mL/min (utilize for a sample injection volume of 25-100mL)
Desorb Temp.:	230˚C to 240˚C
Desorb Flow Rate:	8-10mL/min He
Desorb Time:	3.0 minutes

Refocusing Trap

Temperature:	-180˚C
Injection Temp.:	160˚C
Injection Time:	1.0 min

Adsorbent Trap Reconditioning Conditions

Temperature:	265°C
Initial Bakeout:	2 hours or until clean blank is obtained
After each run:	5-8 minutes

Sample Run Time

Each analytical run is approximately 20 minutes long; the total cycle time is about 30 minutes between injections.

11.5.2 GC/MS System

Optimize GC conditions for compound separation and sensitivity.

Item	Condition
Carrier Gas	Helium
Flow Rate	1.0-1.5mL/minute
Temperature Program	Initial Temperature: ~20˚C Initial Hold Temperature: 3 minutes Ramp Rate: 5˚C/min to 80˚C 2nd Ramp: 10˚C/min to 160˚C 3rd Ramp: 20˚C/min to 240˚C for 5 min hold
Detector B (MSD *Interface)*	260˚C

Electron Energy	70 Volts (nominal)
Mass Range (Scan mode)	34 to 280 amu
Mass Range (SIM mode)	Scan masses corresponding to the target analytes
Scan Time	To give at least 10 scans per peak, not to exceed 1 second per scan.

Note: *The instrument may be operated in Selective Ion Monitoring (SIM) mode if requested by the client.*

11.6 Instrument Performance Check

Since the BFB tuning compound is included in the internal standard and surrogate standard canister and an autosampler is used, it is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to the reduction and approval of any data collection. The 24-hour time period for GC/MS instrument performance check and standards calibration (initial calibration or continuing calibration verification criteria) begins at the injection of the BFB, which shall be documented in laboratory records. Upon completion of the successful BFB tune, the tune report must be printed and retained on file for future reference.

The mass spectrum of BFB must be acquired in the following manner.

- Inject 50ng or less (on column)
- Three scans (peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.
- Background subtraction is conducted using a single scan prior to the elution of BFB.
- All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.
- The ion abundance criteria must not be changed from the requirement stated in this document (TO-15 or TO-14A, as requested).

All subsequent standards, samples and QC samples associated with a BFB analysis must use identical instrument conditions.

11.7 Initial Calibration

The initial calibration is performed to determine instrument sensitivity and the linearity of the GC/MS response for the target compounds.

Initial calibration requirements are as follows:

1. A minimum of 5 concentrations must be used to calculate the calibration curve.
2. An initial calibration must be performed at a minimum initially per instrument, annually thereafter or whenever the continuing calibration verification standard does not meet the acceptance criteria.
3. Highest concentration, together with the lowest concentration, defines the calibration range.
4. The method reporting limit for any reported analyte must be at >/= the lowest calibration point.
5. The initial calibration event may not be interrupted by maintenance.
6. Only one value per concentration may be used.
7. Analyze calibration standards from lowest to highest concentration.

8. All ICAL analyses must be completed within the 24-hour tune window.
9. If 5 calibration standards are in the ICAL, one standard may be re-analyzed. If 6 to 10 calibration standards are in the ICAL, two calibration standards may be re-analyzed.
10. One of the calibration points from the initial calibration curve must be at the same concentration as the continuing calibration verification standard.
11. The upper end of the calibration range must not exhibit any peak saturation for any analyte or the range must be lowered accordingly.
12. The initial calibration model must be linear calibration using average of response factors and cannot be changed for any reason.
13. Point dropping policy
    - Minimum of 5 consecutive concentrations must be used to calculate the calibration curve.
    - Lowest concentration must be at or below the MRL (LOQ) and may not be dropped unless the MRL is changed to the concentration of the remaining lowest standard.
    - Points at the high end may be dropped, but doing so lowers the calibration range.
    - Points may not be dropped from the interior of the curve unless an assignable cause (i.e., gross dilution error, missing internal standards, purge malfunction, standard preparation error, or instrument malfunction) is accounted for and documented. In these instances, all the analytes in that calibration standard must be dropped from the calibration curve as the corrective action (the reason must be documented and the results maintained with the documentation for the final ICAL).
    - Dropping individual compound points from the upper or lower end of the calibration range to improve linearity is not considered an error correction. The reason for dropping these points does not need to be documented but the ICAL documentation must state the revised calibration range if the MRL must be adjusted or the calibration range is lowered for a particular compound. This must be documented on the ICAL Review Checklist.
    - A calibration standard may be re-analyzed if the first analysis of the standard has been dropped and other requirements in this policy are met (i.e., still within 24 hours).
    - Once the ICAL has been used to calculate and report sample results it MUST not to be changed for any reason.
    - It is recommended that if an analyte has a higher MRL than the lowest concentration analyzed that the low standard be automatically dropped from the curve (i.e., acetone MRL is 5, drop at least the 0.5ng point).

11.7.1 <u>Calibration Points</u> Analyze the calibration standards (analyze low to high) that span the monitoring range of interest of the samples. For SCAN, the range is typically 0.5ng-100ng on column; however, 0.1ng on column may be added if low level analyses are requested. For SIM, the range is 10pg on column to 20,000pg on column. The dynamic range is dependent on the sensitivity of a particular instrument as well as the required reporting limit for a given project and may be adjusted accordingly. Refer to Table 3 (SCAN) and Table 3A (SIM) for the concentrations of the compounds of interest in the initial calibration at each particular calibration concentration level.

Note: Refer to the EXCEL TO-15 Standard Concentration templates, located on the network at Q:\\TO15 Std. Concentrations\Std. Conc. Templates for both the SIM and SCAN templates. These templates must be utilized for the documentation of the standard canister concentration selection, final ICAL level concentrations and the determination of the correct injection volumes for the selected standard canister concentrations. If the primary or secondary stock standard cylinder concentrations are revised (upon re-certification or new purchases), the EXCEL spreadsheet templates, injection amounts and the ICAL concentrations in each instrument method must be adjusted accordingly. Other templates may be employed as long as they are validated and provide at least the same information.

SCAN

1. Determine if the lower end of the calibration range is to be 0.1ng or 0.5ng on column. If the low end is 0.1ng, then the 4ng/L standard must be utilized.
2. Determine if the 4ng/L or 20ng/L standard canister is to be used for the 0.5ng on column point.
3. Follow the instructions in the spreadsheet and save the file under the correct instrument folder and the initial calibration method identification.
4. Print the final ICAL concentration sheets and place into the corresponding ICAL folder

11.7.2 Recalibration Each GC/MS system must be recalibrated following any instrument maintenance which may change or effect the sensitivity or linearity of the instrument, if the continuing calibration verification acceptance criteria are not met and at least annually. The following procedure must be followed when updating an initial calibration method.

1. Open the most recent method.
2. Save the method with the new ICAL method ID using the "Save Method As" option. Date used in the method ID must be the date files were analyzed.
3. Quantitate midpoint standard and check retention times and integrations. Update retention times if necessary using QEdit or Easy ID (Tools → Easy ID). Requant if any changes are made and verify all peaks are identified correctly. Print.
   a. While midpoint standard is loaded update reference spectra (Continuing Calibration → Update Reference Spectra).
   b. With midpoint standard loaded update qualifier ion ratios and retention times (Initial Calibration → Update Levels → Select Update Level and then select Retention Times (Replace) and Replace Qualifier Ion Relative Responses).
   c. If necessary adjust integration parameters prior to processing remaining ICAL points.
4. Quantitate remaining ICAL standards. Review each peak for retention time, integration, and print. Review low level standards for acceptable signal to noise ratios and high level standards for saturation.
5. All responses must be cleared from ICAL before updating (Initial Calibration → Clear All Calibration Responses).
6. Update responses for each standard level (Initial Calibration → Update Levels) or (Initial Calibration → Quick Levels Update). If Quick Levels Update is used do not requant datafiles.

7. Save method.
8. Check Response Factor Report and evaluate whether any points should be dropped following the criteria outlined in this SOP.
9. Save method if any changes are made.
10. Verify calibration files listed on Response Factor Report are correct.
11. Verify file ID, acquisition time, quant time, update time, and last update information is correct on the Calibration Status Report.

11.7.3 Analytical Window If time remains in the tune window after meeting the acceptance criteria for the initial calibration, samples may be analyzed according to the procedure described in this document (see Section 11.4.2). If time does not remain in the analytical window, a new sequence shall commence with the analysis of the instrument performance check compound (BFB) and the continuing calibration verification standard.

11.7.4 Procedure The system should be operated using temperature and flow rate parameters equivalent to those in Section 11.5. Use the standard prepared in accordance with Section 9.2.2 of this SOP. Attach the calibration standard and internal standard/surrogate canisters to the designated inlets on the preconcentrator and open the canister valves. Analyzing different volume aliquots of the calibration standards produces differing concentrations.

Analyte responses (target ion areas) are tabulated and recorded using the Enviroquant program. Quantitation ions for the target compounds are shown in Table 2 and 2A and the primary ion should be used unless interferences are present, in which case the secondary ion may be used, but the reason documented in the initial calibration file and all subsequent quantitations utilizing that ICAL must be performed using the same ion selections. Refer to Section 13.3 for the required calculations and Section 12.3 for the acceptance criteria.

11.7.4.1 Additional Requirements The procedure for performing and generating a new initial calibration method must follow a few additional requirements.

1. If any analyte lacks the appropriate sensitivity (3 to 1 signal to noise ratio) at the low end of the calibration range, this point must be dropped from the curve and the MRL/LOQ raised accordingly.
2. No detector saturation may occur for any compound; the upper calibration level must produce no saturated peaks. Exhibited by:
   - The flattening of the response for the higher concentration standards as shown on the plot;
   - The presence of a reverse tail or rise on the front part of the peak;
   - The observed actual percent ratio of the secondary ion presence is lower than the expected percent ratio; or
   - The presence of a flat topped peak and again by the decline or saturation of the secondary ion compared with the expected % recovery.

11.7.4.2 LOQ Establishment, Verification and Acceptance Criteria

1. The LOQ must be set within the calibration range (≥ low std. of the current passing ICAL) prior to sample analysis.
2. The LOQ for each analyte must be > the analyte's LOD.
3. Initially a passing demonstration of precision and bias must be performed at the LOQ.
4. Run CCV 2 times at LOQ and:
   a. Generate a duplicate report for precision using ±25% as the criteria.
   b. Check the %Rec using laboratory generated control limits.
   c. Check the signal to noise ratio (S/N) using the software. The S/N ratio must be at least 3:1 for each analyte.
   d. All ion abundances must be acceptable per the requirements set forth in this document.
5. If any compounds fail, verify at a higher level and notify reporting. Also, make a note in the ICAL documentation.
6. Turn in all LOQ verification data (quant reports and software reports/checks) to QA (regardless of pass/fail).
7. Verify the LOQ on each instrument quarterly.

11.7.5 Initial Calibration Review Analyst's calculation and assessment along with a peer review of all ICAL data and documentation as stated in Attachment B is required before the ICAL may be used to analyze samples. In the case where samples are placed on the autosampler and allowed to run overnight, the sample results may only be reported if the ICAL is reviewed and found to be acceptable. The ICAL checklist in Attachment B must be used to document the review and approval process.

Perform a review of specific aspects of the calibration which might compromise data quality such as inappropriate extension of the calibration range with detector saturation and/or a lack of sensitivity for any analyte. Analyte concentrations which do not meet the signal to noise ratio or exhibit saturation are not to be reported and must be eliminated from the initial calibration. These instances should be followed by a short explanation regarding the reason for the omission.

11.7.6 Initial Calibration File An ICAL file is to be created for each initial calibration performed per instrument into which is placed the following ICAL documents. The file shall remain in the laboratory and be filed by instrument and date.

- ICAL Checklist filled out, reviewed and approved
- BFB tune analysis report
- Calibration status report (aka Calibration History)
- Relative Response Factor Report / Percent Relative Standard Deviation
- Quantitation report for each calibration standard (including manual integration documentation – before and after manual integration)
- ICV quantitation report and % recovery report.
- TO-15 Standard Concentration Spreadsheet (exact ICAL level concentrations and ICV concentrations)
- Any manual integration documentation

11.8 Initial Calibration Verification Standard

Verify the initial calibration by analyzing an initial calibration verification standard (ICV). This standard shall be obtained or prepared from materials acquired from a different manufacturer or lot from that of the initial calibration and prepared according to Section 9.2.3.

Analyze 50ng or less (refer to Table 4 for the secondary source standard concentrations) of the ICV standard depending on the dynamic range of a given instrument and refer to Section 13.5 for the required calculations.

11.9 Continuing Calibration Verification Standard

Verify the calibration each working day, where necessary (e.g., an ICAL was not analyzed or the tune window has closed) by analyzing a continuing calibration verification (CCV) standard from the initial calibration standard canister. The concentration of the calibration verification may be varied between the low calibration standard and the midpoint of the calibration range; however, the concentration must be at one of the levels analyzed in the initial calibration. Refer to Table 3 for the standard concentrations. Refer to Section 13.4 for the required calculations.

11.10 Canister Quality Control Check and Method Blank

The method blank must be a sample of a matrix similar to the batch of associated samples that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure, and in which no target or interferences are present at concentrations that impact the analytical results for sample analyses. Prepare a canister that has not left the building by pressuring with humidified zero air. Analyze an aliquot of one liter along with the same volume of internal standard and surrogate as standards and samples. Additionally, a blank must be analyzed whenever a high concentration sample is encountered and carryover is suspected.

A Quality Control (QC) check canister pressurized with humidified zero air may serve as a method blank as long as the analyte concentration requirements stated in the canister quality control check section (Sections 12.6 and 12.7) and other requirements (refer to Section 12.11 for internal standard requirements) are met. Assuming continuing failure, another QC canister or a new canister must be prepared and analyzed in order to verify that no system contamination exists. For tracking purposes the unique laboratory barcode given to a canister shall be the information included in the sample analysis identification.

11.10.1 Sampling Systems Section 7.1 and 8.4 of Method TO-15 describe the setup and certification procedure for a specific sampling apparatus that has been used by the EPA for several of its large air monitoring programs. These systems are rarely used for the types of projects that make up the bulk of the laboratory's work. The vast majority of samples analyzed by the laboratory are taken into Summa canisters either as grab samples or using a simple time integrated sampling device (flow controller), as in Section 8.2.1 of the method, so these procedures are not part of the typical protocol for providing sampling materials to clients. The laboratory has developed an SOP for the cleaning and certification of the materials it provides its clients for obtaining air samples to be analyzed by method TO-15. Refer to the *SOP for Cleaning and Certification of Summa Canisters and Other Specially Prepared Canisters* for additional information.

It is this laboratory's interpretation that the sampler system certification procedure described in Section 8.4.4 of the TO-15 method applies to the specific sampling apparatus described in the method and not to the sampling procedures used by our clients. The laboratory does not maintain a dynamic calibration manifold or canister sampler apparatus as described in the method and thus performance of the relative accuracy certification procedure described in section 8.4.4. is not possible.

11.11 Laboratory Control Sample

The laboratory control sample is a sample matrix, which is free from the analytes of interest and spiked with a standard containing known amounts of analytes. The laboratory control sample is an injection of the initial calibration verification standard. Inject the LCS (ICV) at concentrations below the midpoint of the calibration curve. Make sure that all of the pertinent information is included on the quantitation report including the sample identification (LCS), concentration, standard used, and analyst.

11.12 Sample Analysis

Prior to analysis, all sample containers (canisters and bags) should be at temperature equilibrium with the laboratory.

- Attach sample canisters to Tekmar AUTOCan using a 9/16" wrench.
- Before opening the valve, check for leaking fittings by running the leak check program in the Teklink software.
- If system is leak tight, open the canister valves and start the automated preconcentration procedure. Make sure the Chemstation data acquisition software has been readied.
- Maintain the trap at an elevated temperature until the beginning of the next analysis.

Check all target compounds using the QEdit routine in Enviroquant, making sure all extracted ion chromatogram peaks are integrated properly (see Section 11.16).

Note: *The secondary ion quantitation is only allowed if there is sample matrix interference with the primary ion. If the secondary ion quantitation is performed, document the reasons in the instrument run logbook and/or on the quantitation report (initial and date any notation).*

SCAN Mode - The instrument is normally operated in the SCAN mode, where the following procedure may be followed.

- Upon sample injection onto the column, the GC/MS system is operated so that the MS scans the atomic range from 34 to 270 amu. At least ten scans per eluting chromatographic peak should be acquired. Scanning allows identification of unknown compounds in the sample through searching of library spectra. See operating conditions in Section 11.5.
- Generate a quantitation report for each run.
- If reporting Tentatively Identified Compounds (TICs), refer to Section 11.12.2 for identification criteria.

SIM Mode - When the client requests SIM mode, select SIM instead of SCAN mode and identify a minimum of two ions per analyte of interest. Also, a minimum of two ions for each internal standard and surrogate compound should be selected.

Helium Pressurization – If a canister is pressurized with helium, a correction factor is applied to sample volumes extracted from the canister via auto sampler. This is due to the difference in thermal properties between helium and air. A correction factor worksheet has been generated to determine the exact volume taken from a canister and may be found at J:\\A-GCMS\Helium Pressurization. Save file, print the sheet and include with the data. Refer to the instruction page in the template for all of the instructions and calculations including backfilled canisters.

AutoCAN Leak Checks – Canisters should be put on at least two different AutoCAN positions to confirm a "leak". In addition, the valve threads should be inspected for defects which may prevent a good seal with the AutoCAN. Once a canister has "failed" the leak check it must be tagged, an NCAR initiated, and the PM notified. Regardless of what the client or PM specifies as the fate of the sample, the canister must be put on maintenance hold to complete a full 24-hour leak check. A yellow sheet is to be completed in addition to, but not in lieu of an NCAR. This is a fixed QA procedure with no allowance for deviation.

11.12.1 Sample Dilution If any target analyte results are above the highest level of the initial calibration, a smaller sample aliquot should be analyzed. The dynamic range of volume aliquots for the automatic cryogenic concentrator is 20cc to 1L. If a volume smaller than 20cc is to be analyzed, a dilution should be made in a Tedlar bag, or the sample directly injected using a gastight syringe. Guidance in performing dilutions and exceptions to this requirement are given below.

- Refer to Section 11.5.1 (Adsorbent Trap Sampling Rate) for the required sampling rate if less than 100mL is to be analyzed.
- Use results of the original analysis to determine the approximate dilution factor required and get the largest analyte peak within the initial calibration range.
- The dilution factor must be documented (and included in the final report) and chosen in such a way as to keep the response of the analyte peak for a reported target compound in the upper half of the initial calibration range of the instrument.

Tedlar bag dilution:
- Make a dilution by filling a Tedlar bag with 1.0 liter of humidified zero air using a one-liter gas syringe.
- Calculate the volume of balance gas needed to obtain the required dilution.
- Remove the difference in the balance gas using a syringe.
- Add the calculated sample amount using a gastight syringe.

Direct injection:
- Make a direct injection by attaching a clean, humidified zero air filled Summa canister to the preconcentrator autosampler using 1/4" stainless steel or teflon tubing with a "tee" septum port. This canister should be the same canister that may be used as the method blank.

- Inject the sample through the septum while the preconcentrator withdraws a 200cc aliquot from the canister.

11.12.2 Tentatively Identified Compounds When requested, a mass spectral library search may be made for the purpose of tentatively identifying sample components not associated with the calibration standards. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system mass spectral library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Certain programs may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. The following guidelines are used for making tentative identifications.

- Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ±20%. For example, for an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30 and 70%.
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.
- The concentration of the tentatively identified compound is estimated by assuming a response factor of 1.0 and comparing the response of the tentatively identified compound to the response of the nearest internal standard.
- If non-target analytes are not Q-deleted from the quant report, the analyst must evaluate whether these compounds should be reported as TICS.

11.13 Duplicate

A duplicate must be analyzed to assess laboratory precision and samples selected for duplicate analysis shall be rotated among client samples, where applicable. Some projects or sample matrix issues may require the analysis of a duplicate laboratory control sample (DLCS).

11.14 Internal Standard (IS)

The concentration of internal standard added to each standard, field sample and QC sample must be consistent from that of each current ICAL standard.

11.15 Surrogates

Internal standards/surrogates must be added at the same volume for every standard, sample and QC sample. Surrogate compound recoveries are requested by a number of clients, but are more appropriately used as system monitoring compounds. This is due to the fact that the compounds are introduced directly into the analytical system and not into the canisters or bags. It is for this reason that they are not considered to be true surrogates and a fixed window is applied. Additionally, surrogates are not included in the ICAL because they are not required by the method and are only system monitoring compounds.

11.16 Manual Integration and Q Deletion

A list of abbreviations (codes) that may be used to give a reason for performing either of these procedures are listed in the *SOP for Data Review and Reporting*.

11.16.1 Manual Integration The integration for each peak must be legally defensible and shall be checked to ensure that it has been integrated properly and consistently between samples, standards and QC samples. All peak reviews and manual integrations must follow the requirements specified in the *SOP for Manual Integration Policy* and the *SOP for Laboratory Ethics and Data Integrity.* The requirements in the above stated procedure include when manual integrations are performed, raw data records shall include a complete audit trail for those manipulations (i.e., chromatograms showing both the integration prior to any manual integrations and those depicting the corresponding manually integrated peaks), and notation of rationale, date, and initials of person performing the manual integration operation. In addition, manual integrations must be reviewed and approved by a second reviewer and the manual integrations maintained in the appropriate job file.

Reporting Requirements Certain project requirements including samples which are submitted under the Department of Defense (DoD) QSM require that the case narrative include an identification of samples and analytes for which manual integration is required. Refer to project requirements to determine if this is necessary.

11.16.2 Q Deletion Q deleting may be performed to either delete a false positive or delete non-target compounds.

11.17 Detection Limits and Limits of Detection

The MDL study shall be performed annually for all target analytes on each instrument (with identical configurations) for which this method is performed. The MDL shall be performed in accordance with the procedure outlined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation.* The detection limit shall be used to determine the LOD for each analyte.

Once determined on each instrument, the highest LOD (for each analyte from all instrument determinations) shall be used as the uniform LOD. However, if a lower detection limit is reported, then the samples must have been run on that specific instrument on which the lower LOD was determined.

11.17.1 Performance and Acceptance Criteria

1. The MDL must be <0.5ppbV for each analyte (Method 11.11.1).
2. Perform Limit of Detection (LOD) verification on all instruments (performing this method) immediately following the MDL study. Spike the LOD at 1-4x the MDL; the spike level establishes the LOD.
3. LOD Acceptance
   - Analyte must be detected reliably and identified by the method-specific criteria (i.e, ion confirmation) and produce a signal that is at least 3 times the instrument's noise level (3:1 signal to noise ratio).
   - It is specific to each combination of analyte, matrix, method and instrument configuration.
   - The LOD must be verified quarterly on each instrument (spiked at LOD) using the criteria listed above.
4. If the LOD verification fails (per #3), repeat the detection limit determination and LOD verification at a higher concentration or perform and pass two consecutive LOD verifications at a higher concentration and set the LOD at the higher concentration.
5. The laboratory shall maintain documentation for all detection limit determinations and LOD verifications (regardless of pass or fail).

11.18 Method Reporting Limit Check Standard

It is recommended to analyze a MRL check standard at the current MRL or required MRL for the batch (per client requirements) of twenty or fewer samples if the CCV fails low for any target compound. A MRL check standard may also be required per client specifications.

This check standard can also serve as the LOQ verification if it meets the specific requirements listed in Section 11.7.4.2. Apply the requirements and retain all documentation accordingly. Refer to Attachment D for Minnesota specified MRL check standard criteria.

11.19 Storing Electronic Data

The initial calibration data must be stored in a quantitation method (on the server) using a unique filename and may not be overwritten at any time in order to maintain an accurate audit trail. There are multiple quantitation methods, which are subsets of the compound list in Table 2. Therefore, files will be named with an eight-character notation indicating the compound list and the date of the corresponding initial calibration. In addition, all data files including method blanks, continuing calibration verification, laboratory control samples and client submitted samples files are saved in a unique sub-directory on the server.

12) Quality Assurance / Quality Control Requirements

12.1 To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

12.2 Instrument Performance Check

Refer to Tables 1 and 1A for the required ion abundance criteria.

12.3 Initial Calibration

- The RRT for each target compound at each calibration level must be within 0.06RRT units of the mean RRT for the compound.
- The calculated %RSD for the RRF for each compound in the calibration standard must be less than 30% with at most two exceptions up to a limit of 40% (this may not be true for all projects).
- For each Internal Standard the area response (Y) at each calibration level must be within 40% of the mean area response $\overline{Y}$ over the initial calibration range.
- The retention time shift for each of the internal standards at each calibration level must be within 20s of the mean retention time over the initial calibration range for each internal standard.
- All of the following information must be retained to permit reconstruction of the initial instrument calibration: calibration date, test method, instrument, analysis date, analyte identification, analyst's initials, concentration and responses, and response factors.
- All initial instrument calibrations must be verified with an acceptable ICV.

12.4 Initial Calibration Verification Standard

The percent recovery for each compound in the ICV must be between 70%-130% for all analytes except vinyl acetate, which must be within 50-150%. Exceptions to this allowance for the vinyl acetate recovery are project specific requirements and any DoD type project, which shall adhere to the 70-130% requirement for all target compounds.

12.5 Continuing Calibration Verification Standard

All compounds must be evaluated prior to rounding. The percent difference for each target analyte must be within plus or minus 30% of the initial calibration average RRFs.

12.6 Canister Quality Control Check

The actual cleaning procedure, number of cans to select for analysis (to release a cleaning batch) and corrective actions are covered in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters* and are not covered in this section. However, the procedure for analyzing and certifying a cleaning batch is included. If a canister passes as a QC canister it meets all of the requirements for a method blank (Method, NELAC\TNI, and Department of Defense Quality Systems Manual – DoD QSM, etc.).

12.6.1 Scan Analyses A canister is considered "clean" for normal SCAN analyses if the analysis shows <0.2ppbv of any target analyte (analyte exceptions listed in table below). If a canister passes as a QC canister it meets all of the requirements for a method blank (Method, NELAC\TNI, and Department of Defense Quality Systems Manual - DoD QSM, etc.).

Low Level SCAN Analyses For those analytes with a MRL of 0.1ug/m3, the QC criteria of <MRL is acceptable; otherwise, <0.2ppbV is required (analyte exceptions listed in table below).

SIM Analyses Results <MRL will be acceptable as this complies with the <0.2ppbV method requirement.

ANALYTE EXCEPTION LIST					
Compounds	ppbV	On Column (ng)	Compounds	ppbV	On Column (ng)
Target Analytes	0.2	0.50	Acrylonitrile	0.2	0.43
Chloromethane	0.2	0.41	Acetone	1.5	3.5
1,3-Butadiene	0.2	0.44	Ethanol	1.9	3.5
Acetonitrile	0.2	0.33	Vinyl acetate	0.99	3.5
Acrolein	0.65	1.5	1-Butanol	0.23	0.70
Isopropanol	0.28	0.70	Carbon Disulfide	1.1	3.5
2-Butanone	1.2	3.5			

Make a notation on the sample identification tag as to the status of the check and return the canister to the canister conditioning room. Additionally, if the check was found to be acceptable, the quantitation report must be kept on file for future reference

12.6.2 Tentatively Identified Compounds (TIC) If the batch of canisters are to be used for tentatively identified compounds (TIC) analysis, any non-target peaks present in the QC check canister analysis must be evaluated and determined to be less than the TIC reporting limit (10% of the internal standard). The concentration is estimated by assuming a RRF of 1.0 and comparing the response of the TIC to the response of the nearest internal standard.

12.7 Method Blank

- The concentration of a targeted analyte in the blank cannot be at or above the MRL, AND be greater than 1/10 of the amount measured in any associated sample. For any project that requires reported results less than the MRL, all associated measurements found in the MB should result in a qualifier; however, project requirements may differ and must be followed. Refer to DoD requirements listed below.
- The method blank should not contain additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte.
- For DoD samples, the method blank will be considered to be contaminated if:

  1. The concentration of any target analyte in the blank exceeds 1/2 the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater);
  2. The concentration of any common laboratory contaminant (acetone, ethanol, carbon disulfide, and methylene chloride) in the blank exceeds the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater); or
  3. The blank result otherwise affects the samples results as per the test method requirements or the project-specific objectives.

The laboratory shall evaluate whether reprocessing of the samples is necessary based on the above criteria.

12.8 Laboratory Control Sample

Round all results to the nearest whole number prior to determining if the acceptance criteria have been met. The percent recoveries must be within the laboratory-generated limits and are referenced in the electronic TO-15 Method Manual. However, Arizona requires the percent recovery for each compound in the LCS to be 70%-130% (to match the ICV requirement). Therefore, the ICV exception for vinyl acetate stated in Section 12.4 requires the percent recovery for AZ samples to be 50-150%.

Note: Client project requirements, AFCEE and DoD requirements shall take precedence over the AZ requirement for AZ samples. Meaning if a sample is collected for a DoD project in AZ, DoD requirements specified in this document and the project specific QAPP (if supplied) are to be followed.

12.9 Sample Results

- Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification.
- Samples out of holding time must be handled according to Section 16.
- The field sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, initial calibration verification technical acceptance criteria described in this document.
- All target analyte peaks must be within the initial calibration range, diluted or reported with the appropriate data qualifier.

12.10 Laboratory Duplicate

The relative percent difference must fall within ±25%. This RPD criterion also applies to duplicate laboratory control samples (DLCS).

12.11 Internal Standards

The following acceptance criteria must be applied to each run (except the ICAL – see Section 12.3).

- The area response for each internal standard in the blank must be within ±40 percent of the area response for each internal standard in the most recent valid calibration. (CCV or mid-point from the initial calibration, whichever is most current).
- The retention time for each internal standard must be within ±0.33 minutes of the retention time for each internal standard in the most recent valid calibration. (CCV or mid-point from the initial calibration, whichever is most current).

12.12 Surrogates

Since the matrix precludes the use of true surrogates and there is no established method criterion, acceptable surrogate recoveries are based on a fixed window of 70 - 130%. This is the typical requirement from clients. Additionally, these limits are referenced in SW-846 for use as guidance in evaluating recoveries. These limits are sufficient for evaluating the effect indicated for the individual sample results.

12.13 Method Reporting Limit Check Standard

Per client requirements or if the CCV is biased low for any compound, then evaluate the MRL check standard. Analyte must be detected reliably and identified by the method-specific criteria (i.e, ion confirmation) and produce a signal that is at least 3 times the instrument's noise level (3:1 signal to noise ratio). Also, a percent difference +/-50% is recommended.

13) Data Reduction and Reporting

13.1 The essential information to be associated with analysis, such as computer data files, run logs, etc. shall include: Sample ID code, date and time (if the holding time is 72 hours) of analysis, instrument operating conditions/parameters (or reference to such data), analysis type, all manual calculations including dilutions and manual integrations, analyst's initials, sample preparation (pressure readings and balance gas if pressurized with helium), standard and reagent origin, receipt, preparation, and use, as well as calibration criteria, frequency and acceptance criteria, data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions.

13.2 This method has specific requirements including the use of canisters; any modification must be reported accordingly. All reports that fall under the laboratory's certificate of approval (in accordance with NELAC/TNI standards) must include a statement(s) clarifying any deviations from the scope of this certification. Refer to Section 13.11 for additional information and specific items, which require this clarification.

13.3 Initial Calibration

Tabulate each of the following:

13.3.1 Equation Number 1 - **Relative Response Factor (RRF):**

$$RRF = \frac{A_x C_{is}}{A_{is} C_x}$$

where:

A_x is the area response of the analyte quantitation ion.
A_{is} is the area response of the corresponding internal standard quantitation ion.
C_{is} Internal standard concentration, ng.
C_x Analyte concentration, ng.

Note: *The equation above is valid under the condition that the volume of internal standard spiking mixture added in all field and QC samples is the same from run to run.*

13.3.2 Equation Number 2 - **Average (or Mean) RRF:**

$$\overline{RRF} = \frac{\sum_{i=1}^{N} RRF_i}{N}$$

where:

RRF_i are the individual RRFs from each concentration level in the initial calibration curve.

N is the number of calibration concentration levels.

13.3.3 Equation Number 3 - **Standard Deviation, SD:**

$$SD = \sqrt{\sum_{i=1}^{N} \frac{\left(RRF_i - \overline{RRF}\right)^2}{N-1}}$$ where:

RRF_i are the individual RRFs from each concentration level in the initial calibration curve.

$\overline{RRF}$ Average (or Mean) RRF of all concentration levels in the initial calibration curve.

N total number of calibration concentration levels

13.3.4 Equation Number 4 - **Percent Relative Standard Deviation, %RSD:**

$$\%RSD = \frac{SD}{\overline{RRF}}(100)$$ where:

SD Standard Deviation calculated in equation number 3

$\overline{RRF}$ Average or Mean RRF

13.3.5 Equation Number 5 - **Relative Retention Time (RRT):**

$$RRT = \frac{RT_C}{RT_{is}}$$ where:

RT_C Retention time of the target compound, seconds.

RT_{is} Retention time of the internal standard, seconds.

13.3.6 Equation Number 6 - **Mean Relative Retention Time ($\overline{RRT}$):**

$$\overline{RRT} = \sum_{i=1}^{n} \frac{RRT_i}{n}$$ where:

$\overline{RRT}$ Mean relative retention time (seconds) for the target compound for all initial calibration levels.

RRT_i Relative retention time for the target compound in level i.

n Number of calibration levels

13.3.7 Equation Number 7 - **Mean Area Response ($\overline{Y}$):**

$$\overline{Y} = \sum_{i=1}^{n} \frac{Y_i}{n}$$ where:

Y_i Area response for the primary quantitation ion for the internal standard for each initial calibration standard.

n number of calibration concentration levels

13.3.8 Equation Number 8 - **Mean Retention Times ($\overline{RT}$):**

$$\overline{RT} = \sum_{i=1}^{n} \frac{RT_i}{n}$$

where:

$\overline{RT}$ Mean retention time, seconds

RT_i Retention time for the internal standard for each initial calibration standard, seconds.

n number of initial calibration levels

13.4 Continuing Calibration Verification

- Calculate the (RRF) of each target compound using equation number 1 (13.3.1).

13.4.1 Equation Number 9 - **Percent Difference, %D:**

$$\%D = \frac{RRFx - \overline{RRF}}{\overline{RRF}}(100)$$

where, for any given analyte:

RRF_x is the RRF from the CCV being evaluated.

$\overline{RRF}$ is the mean RRF from the current calibration curve.

13.5 Percent Recovery – ICV, LCS, Surrogates, MRL Check Standard

13.5.1 Equation Number 10 - **Percent Recovery (%R):**

%R = X/TV x 100

where
X = Concentration of the analyte recovered
TV = True value of amount spiked

13.6 Duplicate Analysis

13.6.1 Equation Number 11 - **Relative Percent Difference (RPD):**

$$\frac{x_1 - x_2}{\overline{x}}(100)$$

where:

x_1 First measurement value
x_2 Second measurement value
$\overline{x}$ Average of the two values

13.7 Internal Standards (IS)

- Calculate the mean area response $\overline{Y}$ for each internal standard using equation number 7 (Section 13.3.7).

- Calculate the mean of the retention times for each internal standard using equation number 8 (Section 13.3.8).

13.8 Pressure Dilution Factor (PDF)

13.8.1 Equation Number 12 - **PDF**, for samples collected in Summa canisters:

$$\text{PDF} = \frac{P_{atm} + P_f}{P_{atm} + P_i}$$ where:

P_{atm} is the ambient atmospheric pressure, 14.7 psi at sea level.
P_f is the final sample canister pressure, in psig.
P_i is the initial sample canister pressure, in psig. This will most often be a negative value (sub-ambient initial pressure).

13.9 Results

If a canister has been pressurized with Helium and the Tekmar AutoCan was utilized, refer to Section 11.12.

13.9.1 Equation Number 13 - For calculating analyte concentrations in a sample, the starting point is the nanogram amount generated by the HP Enviroquant software, which appears on the quantitation report.

$$ng_x = \frac{A_x ng_{is}}{A_{is}\overline{RRF}}$$ where:

ng_x is the nanogram amount of analyte *x.*
A_x is the area response of the analyte's quantitation ion.
A_{is} is the area response of the corresponding internal standard's quantitation ion.
ng_{is} is the internal standard amount, in nanograms.
$\overline{RRF}$ is the average or mean RRFs

13.9.2 Equation Number 14 - The final analyte concentration, C_x, in units of micrograms per cubic meter (μg/m³), is then calculated from the following:

$$C_x = \left(\frac{ng_x PDF}{V}\right)\left(\frac{1\mu g}{1000ng}\right)\left(\frac{1000l}{1m^3}\right)$$ where:

V is the sample volume analyzed, in liters.
PDF is the sample canister pressure dilution factor.

13.9.3 Equation Number 15 - To convert to units of parts per billion volume (ppbv):

$$ppbv = \frac{\mu g/m^3}{MW}x24.46 \qquad \mu g/m^3 = \frac{ppbv}{24.46}xMW$$ where:

MW is the molecular weight (Table 2) of the analyte, in g/mole.

24.46 is the molar volume of an ideal gas at 298 K (25 °C) and 760 mmHg (1 atm), in liters per mole (l/mol).

C_x the final analyte concentration in micrograms per cubic meter.

13.9.4 Equation Number 16 – Helium Pressurization (Injection Amount)

Applicable to canisters pressurized with helium and injected utilizing the mass flow controller of the AutoCAN. For full instructions and calculations, refer to the 1st tab of the template located at: J:\A-GCMS\Helium Pressurization\MFC_GCF _backfill.

13.10 Data Review

The analyst must review data on a real time basis for all calibration and QC data. The QC data must be evaluated by analytical sequence following the data review checklist in Attachment C. The data shall be reviewed and the sample results calculated and assessed by one analyst and reviewed by a second qualified analyst. The data review checklist is used to document the reviews and once it has been completed, initialed and dated it must be filed with each job file.

Initial calibrations must be reviewed in the same manner as QC data with all ICAL documentation retained in a separate file organized by instrument and date. Refer to the initial calibration checklist in Attachment B for the review guideline. The ICAL file must contain all the pertinent information stated in Section 11.7.7.

13.11 Reporting

The results of each test shall be reported clearly, unambiguously and objectively, and shall include all the information necessary for the interpretation of the test results and information required by this laboratory's policy, NELAC\TNI standards, DoD Manual (applicable version, see reference section), client projects, and the TO-15 method including modifications, observances, data qualifiers, and certification information.

If the project requires that results be reported below the MRL (LOQ), but above the LOD all of the requirements specified for normal reporting apply (3:1 S/N ratio and ion abundance). This is regardless of the fact that the results will be qualified as estimated.

13.11.1 Analysis Observations / Case Narrative Summary Form

This form, which is included in the *SOP for Laboratory Storage, Analysis and Tracking*, must be generated when there are specific sample composition information or analysis issues and/or observations. In addition, during the analysis, specific identification information or problems, interferences, calibration issues, flags, and additional/expanded explanation of flags should be added to the form. This form may be modified as long as the sections and basic concepts are reserved. All data qualifiers and flags should follow those listed in the most recent Quality Assurance Manual or as defined in any client requirements.

This form is necessary as a means for documentation. This form, among other information, will be reviewed when compiling the final report and case narrative.

All information regarding the job shall remain in the file, in order that sufficient documentation is available to recreate the job from sample receipt through analysis, data reduction, and reporting.

13.11.2 NELAC\TNI Requirements

The following items do not comply with NELAC\TNI standard requirements and must be reported accordingly. A statement, however worded, must be included in the final report indicating that data reported does not fall under the laboratory's NELAC certificate of approval.

- Reporting any compound which is not included in the second source standard (ICV or LCS) does not meet NELAC requirements.
- In addition, a report that contains a compound not included on the NELAC certificate of approval must also include the statement listed above.

13.11.2.1 Modifications

Method modifications are also not allowed under NELAC\TNI standards; therefore, a statement, however worded, must be included in the final report indicating that data reported does not fall under the laboratory's NELAC certificate of approval. In addition, the following items are considered to be method modifications and must be reported accordingly.

- Sample collection in gas collection bags
- The pressurization of canisters with nitrogen or helium (if EPA Method 3C is requested) refer to Section 11.12.

13.11.3 Surrogates

Only report surrogates at the request of the client. If any surrogate is out of control, all samples results (with surrogates requested) associated with the surrogate must be reported with the appropriate data qualifier.

13.11.4 DoD Requirements

Report results with the appropriate data qualifiers, if samples cannot be reanalyzed for any reason. In addition and at a minimum, the following situations are to be noted in the case narrative: manual integrations, CCV out of control, and results exceeding the calibration range.

14) Method Performance

14.1 An on-going assessment of method performance is conducted in order to ensure that the laboratory is capable of reporting results which are acceptable for its intended use. Validation of the method is confirmed by the examination and provision of objective evidence that these requirements are met.

14.2 Method Detection Limit (MDL)

The procedure used to determine the method detection limits are as stated in the *Code of Federal Regulations* (40 CFR 136 Appendix B) as defined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations are listed in Tables 2 and 2A for both SCAN and SIM modes and were obtained using spiked canisters prepared with humidified zero air, making at least seven replicate measurements of the compounds of interest, computing the standard deviation, and multiplying this value by the appropriate Student's t value for 99 percent confidence. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects. All MDLs, regardless of the mode of operation, meet the method performance criteria of <0.5ppbV.

14.3 Accuracy and Precision

Refer to Section 11.4 in the referenced method for information on replicate precision criteria for method performance. Single laboratory accuracy is presented as the second source initial calibration verification standard, which meets the method performance criteria of 30%. Additionally, laboratory generated control limit data for LCSs are presented for the analytes of interest and may be referenced in the electronic TO-15 Method Manual. Refer to Section 11.7.4.2 for the accuracy and precision requirements for concentrations at the LOQ/MRL.

14.4 Selectivity

Mass spectrometry is considered a more definitive identification technique than single specific detectors such as flame ionization detector (FID), electron capture detector (ECD), photoionization detector (PID), or a multidetector arrangement of these (see discussion in Compendium Method TO-14A). The use of both gas chromatographic retention time and the generally unique mass fragmentation patterns reduce the chances for misidentification.

It is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to initiating any data collection. Upon sample injection onto the column, the GC/MS system is operated so that the MS scans the atomic mass range from 35 to 300 amu. At least ten scans per eluting chromatographic peak must be acquired. Scanning also allows identification of unknown compounds in the sample by searching through library spectra.

The sample analysis using the GC/MS is based in part on a combination of retention times and relative abundances of selected ions. The retention time of each chromatographic peak should be ±0.10 minutes of the library/reference retention time of the compound. The acceptance level for relative abundance should be set at ±20% of the expected abundance. The data should be manually examined by the analyst to determine the reason for the # flag [(#) = qualifier out of range], if present and whether the compound should be reported as found or if there is matrix interference. A background subtraction may aid in this determination. Manual inspection of the qualitative results should also be performed to verify concentrations outside the expected range.

Specific selectivity information is provided in this section and document (such as relative retention time) as well as in the referenced method. Refer to the method for additional information on selectivity.

- Use NIST Library 98 or newer version

- The *reference spectra updates* must be performed with every new ICAL utilizing the mid-level standard (minimum). If needed, the reference spectra may be updated sooner with the continuing calibration standard.
- *Retention time updates* must be performed using EasyID and not by updating to the method (InitCal \ Update Calibration). Refer to the Help selection of the software.

14.5 Demonstration of Capability

This laboratory has continuously performed this method since before July 1999. Therefore, ongoing demonstration of capable shall be performed and documented; however, the initial demonstration of method capability is not required.

14.6 Proficiency Testing (PT) Program

The laboratory shall participate in an air and emissions PT study for TO-15. The testing shall be performed in accordance with this document and meet the frequency and proficiency requirements detailed in the DoD QSM Version 4.2 (Requirement Box 43).

15) Pollution Prevention and Waste Management

15.1 All waste disposals shall be carried out in accordance with the requirements detailed in the *SOP for Waste Disposal.* In addition, canisters must be cleaned in accordance with the requirements detailed in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters.*

16) Corrective Actions for Out-of-Control Data

16.1 Corrective actions shall follow the procedures outlined in the *SOP for Nonconformance and Corrective Action*, where appropriate. Any maintenance which may alter instrument sensitivity or linearity must result in the re-analysis of the entire sequence including the tune compound, ICAL or CCV or any batch QC.

16.2 Tune Does Not Meet Criteria

Perform auto tune or manual tune and then re-analyze BFB. If the BFB acceptance criteria are still not met, the MS must be retuned according to the procedure outlined in the instrument user's manual. Perform necessary maintenance and make notations in the instrument maintenance logbook. It may be necessary to clean the ion source, or quadrupole, or take other necessary actions to achieve the acceptance criteria. An acceptable tune is required for sample results to be calculated and reported.

16.3 Initial Calibration Does Not Meet Calibration Criteria

Follow the initial calibration requirements detailed in Section 11.7 for information on re-analyzing or dropping points and the restriction of maintenance performed during the analysis of the initial calibration standards.

If the initial calibration results are outside the established acceptance criteria, corrective actions must be performed and all associated samples reanalyzed, if reanalysis of the samples is not possible, data associated with an unacceptable initial calibration shall be reported as estimated with the appropriate data qualifiers.

16.4 Initial Calibration Verification Does Not Meet Criteria

If the initial calibration verification technical acceptance criteria are not met, reanalyze and if it fails again, prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column. Perform a new initial calibration if any performed maintenance has altered instrument linearity and/or sensitivity. Perform another initial calibration or if reanalysis is not possible, data associated with an unacceptable ICAL/ICV shall be reported as estimated with the appropriate data qualifiers.

16.5 Continuing Calibration Verification Results Do Not Meet Calibration Criteria

If the continuing calibration verification technical acceptance criteria are not met, reanalyze and if it fails again, prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources of the problem and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column.

If any corrective action and/or reanalysis fails to produce continuing calibration verification within acceptance criteria (analyzed immediately following the initial failure), then either two consecutive successful verifications must be performed following corrective action or a new initial calibration must be performed; however, refer to 16.5.1 below.

16.5.1 Method Reporting Limit Check Standard

If the MRL check standard is unacceptable for any compound (sensitivity; ratio or %D), reanalyze at the same or higher level within the same batch and report data with the CCV flag and case narrative notes accordingly.

16.6 Internal Standards Do Not Meet Criteria

16.6.1 Internal Standard Responses If the problem is with the instrument, perform maintenance. If the problem is with a sample, check for interferences. If the response is high, it is likely that interference is present. In this case, lower the volume or aliquot of the sample and re-analyze. If the problem persists, report the results with the best quality and qualify the results. If the problem is corrected with the lower volume analysis, report those results.

16.6.2 Internal Standard Retention Times If the retention time for any internal standard within the sample changes by more than 20 sec from the latest daily calibration or initial calibration mid-point standard, the GC/MS system must be inspected for malfunctions, and maintenance performed as required. Repeat sample analysis where required.

16.7 Method Blank (MB) Results Do Not Meet Criteria

If the analyte concentration results in the blank do not meet the acceptance criteria repeat analysis with remaining QC canisters until results are acceptable or prepare a canister per Section 11.10. If the analyte results in the blank still do not meet the acceptance criteria the source of the problem must be investigated and measures taken to eliminate the source. Each method blank must be critically evaluated as to the nature of the interference and the effect on the analysis of each sample within the batch.

Determine whether the contamination is from the instrument or due to contamination in the blank container (if results from the new can are not acceptable then the system is probably contaminated). In all cases, the corrective action (reprocessing or data qualifying codes) must be documented. However, the specific corrective action depends on the type of project the blank is utilized for; therefore, refer (below) to the reporting/reprocessing requirements.

DEPARTMENT OF DEFENSE (DoD) QSM PROJECT: Any sample associated with a blank that fails the criteria shall be reprocessed in the same or subsequent analytical batch, except when the sample analysis resulted in a non-detect. If reanalysis is not performed, the results shall be reported with appropriate data qualifier.

OTHER PROJECT TYPE: Appropriate corrective measures must be taken and documented before sample analysis proceeds. However, if this is not a possibility and the results must be reported follow the reporting requirements stated in Section 17.4.

16.8 Laboratory Control Sample (LCS) Results Do Not Meet Criteria

If the LCS criteria are not met, determine whether the cause is instrumentation or the result of a poor injection. If the problem is instrumentation, perform maintenance and if the problem is with the injection re-analyze the LCS. DoD considers the same analyte exceeding the LCS control limits two out of three consecutive LCS to be indicative of non-random behavior; therefore, this trend should be monitored and the appropriate corrective action taken when it occurs.

16.9 Laboratory Duplicate Results Do Not Meet Criteria

If the duplicate results do not meet the technical acceptance criteria, perform another duplicate analysis. If the results are still unacceptable and the associated samples are not reanalyzed then all of the sample results in the associated batch must be flagged accordingly.

16.10 Sample Results Do Not Meet Criteria

- If the retention time for any internal standard within the sample changes by more than 20 sec from the latest daily calibration or initial calibration mid-point standard, the GC/MS system must be inspected for malfunctions, and maintenance performed as required. Repeat sample analysis as needed.
- If the area for any internal standard changes by more than ±40 percent between the sample and the most recent calibration, check for possible matrix interferences and re-analyze at a greater dilution. If the requirement is still not met and matrix interference is not detected the GC/MS system must be inspected for malfunction and maintenance made where necessary.
- When corrective actions are made, samples analyzed while the instrument was not functioning properly must be re-analyzed or the appropriate data qualifiers must be attached to the results.

To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

16.11 Sample Holding Time Expired

The customer is to be notified that the sample's holding time was missed and the customer is to decide if the sample analysis is to continue. The documentation of missed holding time and the client's decision to proceed must be included in the corresponding job file. A statement dictating all holding time occurrences must accompany the sample results in the final report.

16.12 Surrogate Results Do Not Meet Criteria

Poor surrogate recovery should be followed by re-analyzing a smaller aliquot to mitigate any matrix interferences. Evaluate the out of control surrogate for the effect on individual sample results.

17) Contingencies for Handling Out-of-Control or Unacceptable Data

17.1 The following is specific information on how to report unacceptable data. If the data requires a data qualifier flag, as specified in this SOP, refer to Appendix D of the most recent version of the Quality Assurance Manual for the appropriate data qualifier.

17.2 Initial Calibration and/or Initial Calibration Verification

All results reported with an unacceptable ICAL must be reported as estimated and all data shall be reported using defined qualifiers or flags or explained in the case narrative accordingly.

17.3 Continuing Calibration Verification

All results associated with an unacceptable CCV (other than #1 below) must be reported with the appropriate data qualifier, flag and/or explained in the case narrative.

1. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported without a qualifier.
2. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples with detects, then those detects must be reported with a qualifier, flag and/or explained in the case narrative.
3. If however, the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, and there are associated samples that are non-detects, then those non-detects must be reported with qualifiers, flags and/or explained in the case narrative as having less certainty. However, along with the data qualifiers, the case narrative may include information stating the fact that the results were not significantly affected if:
   a. *An MRL check standard was analyzed and found to be acceptable. The MRL must be the same as that analyzed in the MRL check standard for those analytes that were biased low in the CCV. Adjust MRLs (if required), flag data and state the certainty in the case narrative where the sensitivity of the instrument was demonstrated at the MRL; therefore, results were not significantly affected.*
   b. *With the reporting limit adjusted to the next level in the calibration curve (typically 5 times higher) to prove the nonexistence of a false negative and note procedure in case narrative.*
4. If the acceptance criteria was exceeded (biased high) for the CCV and there were detectable results in a sample, the results may be "qualified" if the results exceeded the regulatory/decision limit (this is to be stated in the case narrative along with the data qualifiers or flags).

17.4 Method Blank

- If an analyte in the blank is found to be out of control and the analyte is also found in associated samples, those sample results shall be "flagged" in the report and the method blank results reported.
- If the analyte is found in the blank but not in the sample then the results for the sample may be reported without a qualifier.

17.5 Laboratory Control Sample

All results associated with an out of control laboratory control sample must be reported with the appropriate data qualifier. An indication of whether the LCS was out high or low should also be included.

17.6 Surrogate

Report sample results with the appropriate data qualifier.

17.7 Laboratory Duplicate

All batch sample results associated with an out of control laboratory duplicate must be flagged with the appropriate data qualifier.

17.8 Internal Standard

All target analytes associated with an out of control internal standard must be flagged with the appropriate data qualifier.

17.9 Estimated Sample Results

17.9.1 Sample Hold Time All occurrences of missed holding times must be included on the final report including those samples received and/or analyzed outside of the specified hold times detailed in this SOP.

17.9.2 Matrix Interference Sample data associated with matrix interference must be flagged with the appropriate data qualifier.

17.9.3 Results Outside Initial Calibration Range All sample results not bracketed by initial calibration standards (within calibration range) must be reported as having less certainty by reporting with the appropriate data qualifier.

18) Training

18.1 Demonstration of Capability

All analysts must be trained in accordance with the guidelines detailed in the *SOP for Training Policy*. Demonstrations shall also be performed in accordance with the 2009 TNI Standards (Volume 1 Module 4 Section 1.6) and DoD Quality Systems Manual 4.2 (Requirement Box 25). Attachment A shall be used to document the training plan for new analysts' initial demonstration. Additionally, these demonstrations are performed anytime there is a change in instrument type, personnel or method.

Once performance is found to be acceptable, a required certification statement must be completed by the QA Manager and either the immediate supervisor or Laboratory Manager and retained on file as a demonstration of compliance.

18.1.1 Quarterly Demonstration A demonstration of method sensitivity must be performed *quarterly on each instrument* performing this method.

1) A spike at the current LOD must be analyzed.
2) Verification of precision and bias at the LOQ must be performed.

Refer to Section 11.7.4.2 (LOQ) and 11.17.1 (LOD) for additional information on how these demonstrations are to be performed as well as the acceptance criteria.

18.1.2 Annual Demonstration Each analyst must perform this demonstration both initially and annually. Analyze four LCS standards at 1-4x the MRL (LOQ) either concurrently or over a period of days as a verification of precision and bias of the quantitation range. The standard deviation (n-1) and average percent recovery of the four replicates are compared against the method requirement for precision (±25%) and current laboratory control limits for bias/LCS.

18.1.3 Change in Personnel, Instruments, Method and/or Matrix The requirements in Sections 18.1.1 and 18.1.2 must be performed per the schedule noted and when there is a change in personnel, instruments, method or matrix. "Change" refers to any change in personnel, instrument, test method, or sample matrix that potentially affects the precision and bias, sensitivity, or selectivity of the output (e.g., a change in the detector, column type, matrix, or other components of the sample analytical system, or a method revision).

All completed attempts at this demonstration must be completed and turned into the QA department for retention.

19) Method Modifications

19.1 Method modifications are not allowed under NELAC\TNI standards; therefore, a statement, however worded, must be included in the final report indicating that data reported does not fall under the laboratory's NELAC certificate of approval. In addition, the following items are considered to be method modifications and must be reported accordingly.

- Sample collection in gas collection bags
- The pressurization of canisters with nitrogen or helium (if EPA Method 3C is requested) refer to Section 11.11.

20) Summary of Changes

20.1 SOP updated using ALS SOP Template – New cover page, document tracking/signature page, and table of contents page. Header/footer and font revised throughout SOP to align with ALS SOP template specifications. Sections reorganized as outlined in the *SOP for Establishing Standard Operating Procedures* and section references updated as necessary. Section titled "Instrument Specific Addendum" removed (Section 21.0 in previous revision - "Not Applicable" listed under section).

Table of Contents	Updated
Section 1	Renamed section heading "Scope and Applicability"
Section 2	Renamed section heading "Summary of Procedure"
Section 4	Was Section 10 in previous revision
Section 5	Was Section 4 in previous revision
Section 6	Was Section 5 in previous revision
Section 7	Was Section 6 in previous revision
Section 8	Was Section 7 in previous revision
Section 8.5	First Bullet – Updated Column
Section 8.6	Added Windows 7
Section 9	Was Section 8 in previous revision
Section 10	Was Section 9 in previous revision
Section 11.1	Updated SOP title
Section 11.5.1	Edited setpoints to reflect current use
Section 11.7	#13 – 4th bullet – Added parentheses in last sentence #13 – 5th bullet – Added for clarification
Section 11.10.1	Added second paragraph
Section 11.12.1	Updated "25cc" to "20cc"
Section 11.16.1	Updated both SOP titles Removed reference to SOP attachment
Section 13.9.4	Updated file path
Section 16.1	Updated SOP title
Section 18	Was Section 19 in previous revision Renamed section heading "Training"
Section 18.1	Updated SOP title Updated "QA Program Manager" to "QA Manager"
Section 19	Was Section 20 in previous revision
Section 20	Was Section 22 in previous revision
Table 2	Updated MDL values Corrected m-,p-Xylenes CAS Number
Table 2A	Updated MDL values
Table 3	Updated values
Table 3A	Updated values
Table 4	Updated values
Table 4A	Updated values
Attachment A	Removed information at top of checklist covered in SOP header #4 – Updated 2nd, 3rd, and 5th SOP titles
Attachment B	Removed MS10 from list of instruments and added MS21 #13 – Changed "Point" to "calibration level"
Attachment C	Revised Note in Parentheses at top of checklist Changed "CAS Project #" to "Project #" Removed MS10 from list of instruments and added MS21

21) References

21.1 EPA Method TO-14A, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/625/R-96/010b, U.S. Environmental Protection Agency, Research Triangle Park, NC, January 1997.

21.2 EPA Method TO-15, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/625/R-96/010b, U.S. Environmental Protection Agency, Research Triangle Park, NC, January 1997.
21.3 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, January 1999.
21.4 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Addendum, January 17, 2002.
21.5 National Environmental Laboratory Accreditation Conference, *2003 NELAC Standard*, June 5, 2003, EPA 600/R-04/003 and 2009 TNI Standards.
21.6 *Preparation of Gas Phase Standards for Ambient Air Analysis,* Tekmar-DOHRMANN Application Note, Spring 96, Vol. 6.5.
21.7 *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 4.2, 10/25/2010.
21.8 Arizona Administrative Code, Title 9. Health Services, Chapter 14. Department of Health Services Laboratories, December 31, 2006.
21.9 Florida Department of Environmental Protection, Chapter 62-160.
21.10 Minnesota Department of Health, 4740.2065, *Standard Operating Procedures*, Statutory Authority: MS s 144.97; 144.98; History: 31 SR 446, Posted: October 09, 2006, Revised April 16, 2010.

22) Attachments

22.1 Tables

Table 1: Instrument Tune Check Ion Abundance Criteria (TO-15)
Table 1A: Instrument Tune Check Ion Abundance Criteria (TO-14A)
Table 2: Volatile Organic Compounds, EPA Compendium Method TO-15 (SCAN)
Table 2A: Volatile Organic Compounds, EPA Compendium Method TO-15 (SIM)
Table 3: Standard Concentrations (SCAN) (Primary Sources)
Table 3A: Standard Concentrations (SIM) (Primary Sources)
Table 4: Standard Concentrations (SCAN) (Secondary Sources)
Table 4A: Standard Concentrations (SIM) (Secondary Sources)

22.2 Attachments

Attachment A: Training Plan for Analysis of TO-15 by GC/MS
Attachment B: TO-15 by GC/MS Initial Calibration Checklist
Attachment C: TO-15 by GC/MS Analyses Data Review Checklist
Attachment D: State and Project Specific Requirement

TABLE 1

Required BFB Key Ions and
Ion Abundance Criteria for Method TO-15

Mass	Ion Abundance Criteria[1]
50	8.0 to 40.0 percent of m/e 95
75	30.0 to 66.0 percent of m/e 95
95	Base Peak, 100 Percent Relative Abundance
96	5.0 to 9.0 Percent of m/e 95
173	Less than 2.0 Percent of m/e 174
174	50.0 to 120.0 Percent of m/e 95
175	4.0 to 9.0 Percent of m/e 174
176	93.0 to 101.0 Percent of m/e 174
177	5.0 to 9.0 Percent of m/e 176

[1] All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

TABLE 1A

Required BFB Key Ions and
Ion Abundance Criteria for Method TO-14A

Mass	Ion Abundance Criteria
50	15 to 40 percent of m/e 95
75	30 to 60 percent of m/e 95
95	Base Peak, 100 Percent Relative Abundance
96	5 to 9 Percent of m/e 95
173	Less than 2 Percent of m/e 174
174	>50 Percent of m/e 95
175	5 to 9 Percent of m/e 174
176	>95 and <101 Percent of m/e 174
177	5 to 9 Percent of m/e 176

Note: The criteria listed in Tables 1 and 1A shall be met or exceeded in order for EPA Compendium Methods TO-15 or TO-14A to be referenced.

TABLE 2 - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Bromochloromethane (IS1)	74-97-5	-	-	130	128, 132	-	-	-
Propene	115-07-1	42.08	NA	42	39,41	0.50	0.13	IS1
Dichlorodifluoromethane (CFC 12)[5]	75-71-8	120.9	1.329	85	87, 101, 103	0.50	0.14	IS1
Chloromethane	74-87-3	50.49	0.911	50	52	0.50	0.15	IS1
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	76-14-2	170.9	1.455	135	137	0.50	0.14	IS1
Vinyl Chloride	75-01-4	62.50	0.9106	62	64	0.50	0.15	IS1
1,3-Butadiene	106-99-0	54.09	0.6149	54	39, 53	0.50	0.16	IS1
Bromomethane	74-83-9	94.94	1.6755	94	96	0.50	0.14	IS1
Chloroethane	75-00-3	64.52	0.8902	64	66	0.50	0.14	IS1
Ethanol	64-17-5	46.07	0.7893	45	46	5.0	0.92	IS1
Acetonitrile	75-05-8	41.05	0.7857	41	40	0.50	0.24	IS1
Acrolein	107-02-8	56.06	0.840	56	55	2.0	0.20	IS1
Acetone	67-64-1	58.08	0.7845	58	43	5.0	1.1	IS1
Trichlorofluoromethane	75-69-4	137.4	NA	101	103	0.50	0.13	IS1
Isopropyl Alcohol	67-63-0	60.10	0.7809	45	43	5.0	0.34	IS1
Acrylonitrile	107-13-1	53.06	0.8060	53	52	0.50	0.18	IS1
1,1-Dichloroethene	75-35-4	96.94	1.213	96	61	0.50	0.16	IS1
tert-Butanol	75-65-0	74.12	0.7887	59	57,41,43	1.0	0.29	IS1
Methylene Chloride	75-09-2	84.94	1.3266	84	49	0.50	0.15	IS1
Allyl Chloride	107-05-1	76.53	0.9376	41	76	0.50	0.14	IS1
Trichlorotrifluoroethane	76-13-1	187.38	1.5635	151	101	0.50	0.20	IS1

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	CAS Number	Molecular Weight	Density	Primary Ion[2]	Secondary Ion(s)[2]	MRL[3] (µg/m³)	MDL[3] (µg/m³)	IS[4]
Carbon Disulfide	75-15-0	76.14	1.2632	76	78	5.0	0.13	IS1
trans-1,2-Dichloroethene	156-60-5	96.94	1.2565	61	96	0.50	0.15	IS1
1,1-Dichloroethane	75-34-3	98.96	1.1757	63	65	0.50	0.14	IS1
Methyl tert-Butyl Ether	1634-04-4	88.15	0.7402	73	57	0.50	0.14	IS1
Vinyl Acetate	108-05-4	86.09	0.9317	86	43	5.0	0.60	IS1
2-Butanone (MEK)	78-93-3	72.11	0.7999	72	43	5.0	0.20	IS1
cis-1,2-Dichloroethene	156-59-2	96.94	1.2837	61	96	0.50	0.16	IS1
Diisopropyl Ether	108-20-3	102.18	0.7241	87	45,59,43	0.50	0.16	IS1
Ethyl Acetate	141-78-6	88.106	0.9003	61	70	1.0	0.32	IS1
n-Hexane	110-54-3	86.18	0.6548	57	86	0.50	0.16	IS1
Chloroform	67-66-3	119.4	1.4832	83	85	0.50	0.14	IS1
1,2-Dichloroethane-d4(S)	17060-07-0	-	-	65	67	-	-	IS1
Tetrahydrofuran	109-99-9	72.11	0.8892	72	71,42	0.50	0.26	IS1
Ethyl tert-Butyl Ether	637-92-3	102.176	0.7519	87	59,57	0.50	0.15	IS1
1,2-Dichloroethane	107-06-2	98.96	1.2351	62	64	0.50	0.14	IS1
1,4-Difluorobenzene(IS2)	540-36-3	-	-	114	88	-	-	-
1,1,1-Trichloroethane	71-55-6	133.4	1.3390	97	99, 61	0.50	0.14	IS2
Isopropyl acetate	108-21-4	102.13	0.8718	61	87,43	1.0	0.29	IS2
1-Butanol	71-36-3	74.1224	0.8098	56	41	1.0	0.43	IS2
Benzene	71-43-2	78.11	0.8765	78	77	0.50	0.14	IS2
Carbon Tetrachloride	56-23-5	153.8	1.5940	117	119	0.50	0.15	IS2

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion**[2]	**Secondary Ion(s)**[2]	**MRL**[3] **(µg/m^3)**	**MDL**[3] **(µg/m^3)**	**IS**[4]
Cyclohexane	110-82-7	84.16	0.7739	84	69,56	1.0	0.26	IS2
tert-Amyl Methyl Ether	994-05-8	102.176	0.7703	73	87,55,43	0.50	0.15	IS2
1,2-Dichloropropane	78-87-5	113	1.1560	63	62	0.50	0.14	IS2
Bromodichloromethane	75-27-4	163.8	1.980	83	85	0.50	0.15	IS2
Trichloroethene	79-01-6	131.4	1.4642	130	132	0.50	0.14	IS2
1,4-Dioxane	123-91-1	88.11	1.0337	88	58	0.50	0.14	IS2
Isooctane	540-84-1	114.23	0.6877	57	41	0.50	0.13	IS2
Methyl Methacrylate	80-62-6	100.12	0.944	100	69	1.0	0.26	IS2
n-Heptane	142-82-5	100.2	0.6837	71	57,100	0.50	0.14	IS2
cis-1,3-Dichloropropene	10061-01-5	111	1.224	75	77	0.50	0.13	IS2
4-Methyl-2-Pentanone	108-10-1	100.2	0.7965	58	85	0.50	0.16	IS2
trans-1,3-Dichloropropene	10061-02-6	111	1.217	75	77	0.50	0.13	IS2
1,1,2-Trichloroethane	79-00-5	133.4	1.4397	97	83	0.50	0.13	IS2
Chlorobenzene-d5(IS3)	3114-55-4	-	-	82	117	-	-	-
Toluene-d8(S)	2037-26-5	-	-	98	100	-	-	IS3
Toluene	108-88-3	92.14	0.8669	91	92	0.50	0.14	IS3
2-Hexanone	591-78-6	100.16	0.8113	43	58	0.50	0.19	IS3
Dibromochloromethane	124-48-1	208.3	2.451	129	127	0.50	0.15	IS3
1,2-Dibromoethane	106-93-4	187.9	2.1791	107	109	0.50	0.15	IS3
n-Butyl Acetate	123-86-4	116.16	0.8825	43	56, 73	0.50	0.18	IS3
n-Octane	111-65-9	114.23	0.6986	57	114	0.50	0.14	IS3

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)

Compound[1]	CAS Number	Molecular Weight	Density	Primary Ion[2]	Secondary Ion(s)[2]	MRL[3] ($\mu g/m^3$)	MDL[3] ($\mu g/m^3$)	IS[4]
Tetrachloroethene	127-18-4	165.8	1.6227	166	164	0.50	0.12	IS3
Chlorobenzene	108-90-7	112.6	1.1058	112	114	0.50	0.15	IS3
Ethylbenzene	100-41-4	106.2	0.8670	91	106	0.50	0.15	IS3
m-, p-Xylenes	179601-23-1	106.2	0.8642, 0.8611	91	106	1.0	0.29	IS3
Bromoform	75-25-2	252.8	2.899	173	175	0.50	0.16	IS3
Styrene	100-42-5	104.1	0.9060	104	78, 103	0.50	0.15	IS3
o-Xylene	95-47-6	106.2	0.8802	91	106	0.50	0.16	IS3
n-Nonane	111-84-2	128.26	0.7176	43	57, 85	0.50	0.14	IS3
1,1,2,2-Tetrachloroethane	79-34-5	167.9	1.5953	83	85	0.50	0.15	IS3
4-Bromofluorobenzene(S)	460-00-4	-	-	174	176	-	-	IS3
Cumene	98-82-8	120.2	0.8618	105	120	0.50	0.14	IS3
alpha-Pinene	80-56-8	136.24	0.8582	93	77	0.50	0.14	IS3
n-Propylbenzene	103-65-1	120.1938	0.8670	91	120,65	0.50	0.17	IS3
3-Ethyltoluene	620-14-4	120.2	0.8645	105	120	0.50	0.16	IS3
4-Ethyltoluene	622-96-8	120.2	0.8614	105	120	0.50	0.15	IS3
1,3,5-Trimethylbenzene	108-67-8	120.2	0.8652	105	120	0.50	0.15	IS3
alpha-Methylstyrene	98-83-9	118.19	0.9106	118	103,117	0.50	0.21	IS3
2-Ethyltoluene	611-14-3	120.2	0.8807	105	120	0.50	0.15	IS3
1,2,4-Trimethylbenzene	95-63-6	120.2	0.8758	105	120	0.50	0.15	IS3
n-Decane	124-18-5	142.28	0.7300	57	71,85	0.50	0.15	IS3
Benzyl Chloride	100-44-7	126.59	1.1004	91	126	0.50	0.15	IS3

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
1,3-Dichlorobenzene	541-73-1	147	1.2884	146	148	0.50	0.14	IS3
1,4-Dichlorobenzene	106-46-7	147	1.2475	146	148	0.50	0.15	IS3
sec-Butylbenzene	135-98-8	134.2206	0.8601	105	134,91	0.50	0.16	IS3
p-Isopropyltoluene	99-87-6	134.2206	0.8573	119	134,91	0.50	0.16	IS3
1,2,3-Trimethylbenzene	526-73-8	120.1938	0.8944	105	120	0.50	0.15	IS3
1,2-Dichlorobenzene	95-50-1	147	1.3059	146	148	0.50	0.16	IS3
d-Limonene	5989-27-5	136.24	0.8402	68	93	0.50	0.17	IS3
1,2,Dibromo-3-Chloropropane	96-12-8	236.33	2.093	157	75, 39	0.50	0.14	IS3
n-Undecane	1120-21-4	156.31	0.7402	57	71, 85	0.50	0.15	IS3
1,2,4-Trichlorobenzene	120-82-1	181.5	1.459	180	182, 184	0.50	0.18	IS3
Naphthalene	91-20-3	128.17	1.0253	128	129	0.50	0.21	IS3
n-Dodecane	112-40-3	170.34	0.7487	57	71,85	0.50	0.21	IS3
Hexachlorobutadiene	87-68-3	260.8	1.556	225	227	0.50	0.14	IS3
Cyclohexanone	108-94-1	98.14	0.9478	55	42, 98	0.50	0.19	IS3
tert-Butylbenzene	98-06-6	134.22	0.867	119	134	0.50	0.16	IS3
n-Butylbenzene	104-51-8	134.22	0.867	91	134	0.50	0.16	IS3

(S) = Surrogate (IS1) = Internal Standard 1 (IS2) = Internal Standard 2 (IS3) = Internal Standard 3
NA = Not Available

Note 1: Additional compounds may be reported as long as the minimum requirements of this document are met. The compounds listed in this table are reported using TO-15 SCAN. The Selected Ion Monitoring (SIM) compounds are a subset of this list and are included in Table 2A.

Note 2: These are suggested primary and secondary ions. However, any ions in the analyte spectra that are sufficient enough in response to reach the desired reporting limit and having a limited amount of interference, is acceptable for both the primary and secondary ion selection. Analyst experience should be utilized in determining appropriate ions.

Note 3: The laboratory performs three concentration level analyses (SIM, SCAN and Low Level SCAN). The method reporting limit listed is the standard SCAN limit (at or above lowest concentration in the initial calibration curve), but may change with each new initial calibration performed. Therefore, current reporting limits for the three analysis levels, MRLs in ppbv, and those from the Low Level SCAN should be reviewed in the electronic TO-15 Method Manual.

Note 4: The listing of the internal standard by which the compounds are quantitated is for TO-15 SCAN only. SIM compounds (SCAN subset) and their corresponding ions and internal standards are listed in Table 2A.

Note 5: m/e 101 is ~10% or less of m/e 85 (the base peak) and may not be present for low level results. Retention times must be carefully verified.

Table 2A – Volatile Organic Compounds, EPA Compendium Method TO-15 (SIM)					
Compound	**Primary Ion[1]**	**Secondary Ion[1]**	**MRL[2] (ug/m3)**	**MDL[2] (ug/m3)**	**IS**
Dichlorodifluoromethane	85	87	0.025	0.0024	IS1
Chloromethane	52	50	0.025	0.0031	IS1
Vinyl Chloride	62	64	0.025	0.0025	IS1
Bromomethane	94	96	0.025	0.0052	IS1
Chloroethane	64	66	0.025	0.0021	IS1
Acetone*	58	43	0.50	NA	IS1
Freon 11	101	103	0.025	0.0025	IS1
1,1-Dichloroethene	96	98,61	0.025	0.0031	IS1
Methylene Chloride	84	49	0.10	0.0095	IS1
Trichlorotrifluoroethane	151	153	0.025	0.0030	IS1
trans-1,2-Dichloroethene	96	98,61	0.025	0.011	IS1
1,1-Dichloroethane	63	65	0.025	0.0023	IS1
Methyl tert-Butyl Ether*	73	57	0.025	0.0038	IS1
cis-1,2-Dichloroethene	96	98,61	0.025	0.0098	IS1
Chloroform	83	85	0.10	0.0031	IS1
1,2-Dichloroethane	62	64	0.025	0.0087	IS1
1,1,1-Trichloroethane	97	99	0.025	0.0032	IS1
Benzene	78	77	0.075	0.0046	IS1
Carbon Tetrachloride	117	119	0.025	0.0029	IS1
1,2-Dichloropropane	63	62,76	0.025	0.0021	IS2
Bromodichloromethane	83	85	0.025	0.0036	IS2
Trichloroethene	130	132	0.025	0.0058	IS2
1,4-Dioxane*	88	58	0.10	0.0048	IS2
cis-1,3-Dichloropropene	75	77,39	0.025	0.0095	IS2
trans-1,3-Dichloropropene	75	77,39	0.025	0.015	IS2
1,1,2-Trichloroethane	83	97,61	0.10	0.0034	IS2
Toluene	91	92	0.10	0.0031	IS2
1,2-Dibromoethane	107	109	0.025	0.013	IS2
Tetrachloroethene	166	164	0.025	0.0028	IS2
Chlorobenzene	112	114	0.10	0.0066	IS3
Ethylbenzene	91	106	0.10	0.0046	IS3
m-&-p-Xylene	91	106	0.10	0.0097	IS3
o-Xylene	91	106	0.10	0.0044	IS3
1,1,2,2-Tetrachloroethane	83	85	0.025	0.0047	IS3
1,3-Dichlorobenzene	146	148	0.025	0.011	IS3
1,4-Dichlorobenzene	146	148	0.025	0.012	IS3
1,2-Dichlorobenzene	146	148	0.025	0.0087	IS3
1,2,4-Trichlorobenzene	182	184	0.025	0.019	IS3
Naphthalene	128	129	0.10	0.018	IS3
Hexachlorobutadiene*	225	227	0.025	0.0092	IS3

* Reported upon request NA = Not Available
(IS1) = Internal Standard 1 (IS2) = Internal Standard 2 (IS3) = Internal Standard 3

Note 1: These are suggested primary and secondary ions. However, any ions in the analyte spectra that is sufficient enough in response to reach the desired reporting limit and having a limited amount of interference, is acceptable for both the primary and secondary ion selection. Analyst experience should be utilized in determining appropriate ions.

Note 2: The method reporting limit listed is the standard SIM limit (lowest concentration in the initial calibration curve; must be higher than MDL), but may change with each new initial calibration performed. Therefore, current reporting limits should be reviewed. MDLs in ppbV may be reviewed in the electronic TO-15 Method Manual.

Table 3
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.1ng	0.2ng	0.5ng	1.0ng	5.0ng	25ng	50ng	100ng
Bromochloromethane (IS1)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Propene	0.104	0.208	0.520	1.04	5.20	26.00	52.0	104
Dichlorodifluoromethane (CFC 12)	0.102	0.204	0.510	1.02	5.10	25.50	51.0	102
Chloromethane	0.099	0.198	0.495	0.99	4.95	24.75	49.5	99
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
Vinyl Chloride	0.100	0.200	0.500	1.00	5.00	25.00	50.0	100
1,3-Butadiene	0.122	0.244	0.610	1.22	6.10	30.50	61.0	122
Bromomethane	0.100	0.200	0.500	1.00	5.00	25.00	50.0	100
Chloroethane	0.100	0.200	0.500	1.00	5.00	25.00	50.0	100
Ethanol	0.471	0.942	2.355	4.71	23.55	117.75	235.5	471
Acetonitrile	0.112	0.224	0.560	1.12	5.60	28.00	56.0	112
Acrolein	0.104	0.208	0.520	1.04	5.20	26.00	52.0	104
Acetone	0.539	1.078	2.695	5.39	26.95	134.75	269.5	539
Trichlorofluoromethane	0.102	0.204	0.510	1.02	5.10	25.50	51.0	102
Isopropyl Alcohol	0.189	0.378	0.945	1.89	9.45	47.25	94.5	189
Acrylonitrile	0.112	0.224	0.560	1.12	5.60	28.00	56.0	112
1,1-Dichloroethene	0.109	0.218	0.545	1.09	5.45	27.25	54.5	109
tert-Butanol	0.209	0.418	1.045	2.09	10.45	52.25	104.5	209
Methylene Chloride	0.107	0.214	0.535	1.07	5.35	26.75	53.5	107
Allyl Chloride	0.108	0.216	0.540	1.08	5.40	27.00	54.0	108
Trichlorotrifluoroethane	0.107	0.214	0.535	1.07	5.35	26.75	53.5	107
Carbon Disulfide	0.104	0.208	0.520	1.04	5.20	26.00	52.0	104
trans-1,2-Dichloroethene	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
1,1-Dichloroethane	0.104	0.208	0.520	1.04	5.20	26.00	52.0	104
Methyl tert-Butyl Ether	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
Vinyl Acetate	0.492	0.984	2.460	4.92	24.60	123.00	246.0	492
2-Butanone (MEK)	0.110	0.220	0.550	1.10	5.50	27.50	55.0	110
cis-1,2-Dichloroethene	0.108	0.216	0.540	1.08	5.40	27.00	54.0	108
Diisopropyl Ether	0.109	0.218	0.545	1.09	5.45	27.25	54.5	109
Ethyl Acetate	0.214	0.428	1.070	2.14	10.70	53.50	107.0	214
n-Hexane	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
Chloroform	0.108	0.216	0.540	1.08	5.40	27.00	54.0	108
1,2-Dichloroethane-d4 (S)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Tetrahydrofuran	0.109	0.218	0.545	1.09	5.45	27.25	54.5	109
Ethyl tert-Butyl Ether	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
1,2-Dichloroethane	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
1,4-Difluorobenzene(IS2)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
1,1,1-Trichloroethane	0.103	0.206	0.515	1.03	5.15	25.75	51.5	103
Isopropyl acetate	0.218	0.436	1.090	2.18	10.90	54.50	109.0	218
1-Butanol	0.205	0.410	1.025	2.05	10.25	51.25	102.5	205

Table 3 - Continued
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.1ng	0.2ng	0.5ng	1.0ng	5.0ng	25ng	50ng	100ng
Benzene	0.110	0.220	0.550	1.10	5.50	27.50	55.0	110
Carbon Tetrachloride	0.110	0.220	0.550	1.10	5.50	27.50	55.0	110
Cyclohexane	0.208	0.416	1.040	2.08	10.40	52.00	104.0	208
tert-Amyl Methyl Ether	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
1,2-Dichloropropane	0.103	0.206	0.515	1.03	5.15	25.75	51.5	103
Bromodichloromethane	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
Trichloroethene	0.103	0.206	0.515	1.03	5.15	25.75	51.5	103
1,4-Dioxane	0.108	0.216	0.540	1.08	5.40	27.00	54.0	108
Isooctane	0.103	0.206	0.515	1.03	5.15	25.75	51.5	103
Methyl Methacrylate	0.210	0.420	1.050	2.10	10.50	52.50	105.0	210
n-Heptane	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
cis-1,3-Dichloropropene	0.099	0.198	0.495	0.99	4.95	24.75	49.5	99
4-Methyl-2-Pentanone	0.109	0.218	0.545	1.09	5.45	27.25	54.5	109
trans-1,3-Dichloropropene	0.111	0.222	0.555	1.11	5.55	27.75	55.5	111
1,1,2-Trichloroethane	0.102	0.204	0.510	1.02	5.10	25.50	51.0	102
Chlorobenzene-d5 (IS3)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Toluene-d8 (S)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Toluene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
2-Hexanone	0.120	0.240	0.600	1.20	6.00	30.00	60.0	120
Dibromochloromethane	0.113	0.226	0.565	1.13	5.65	28.25	56.5	113
1,2-Dibromoethane	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
n-Butyl Acetate	0.119	0.238	0.595	1.19	5.95	29.75	59.5	119
n-Octane	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
Tetrachloroethene	0.096	0.192	0.480	0.96	4.80	24.00	48.0	96
Chlorobenzene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
Ethylbenzene	0.104	0.208	0.520	1.04	5.20	26.00	52.0	104
m- & p-Xylene	0.204	0.408	1.020	2.04	10.20	51.00	102.0	204
Bromoform	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
Styrene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
o-Xylene	0.101	0.202	0.505	1.01	5.05	25.25	50.5	101
n-Nonane	0.103	0.206	0.515	1.03	5.15	25.75	51.5	103
1,1,2,2-Tetrachloroethane	0.098	0.196	0.490	0.98	4.90	24.50	49.0	98
4-Bromofluorobenzene (S)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Cumene	0.101	0.202	0.505	1.01	5.05	25.25	50.5	101
alpha-Pinene	0.098	0.196	0.490	0.98	4.90	24.50	49.0	98
n-Propylbenzene	0.101	0.202	0.505	1.01	5.05	25.25	50.5	101
3-Ethyltoluene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
4-Ethyltoluene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
1,3,5-Trimethylbenzene	0.107	0.214	0.535	1.07	5.35	26.75	53.5	107
alpha-Methylstyrene	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
2-Ethyltoluene	0.103	0.206	0.515	1.03	5.15	25.75	51.5	103
1,2,4-Trimethylbenzene	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105

Table 3 - Continued
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.1ng	0.2ng	0.5ng	1.0ng	5.0ng	25ng	50ng	100ng
n-Decane	0.103	0.206	0.515	1.03	5.15	25.75	51.5	103
Benzyl Chloride	0.107	0.214	0.535	1.07	5.35	26.75	53.5	107
1,3-Dichlorobenzene	0.107	0.214	0.535	1.07	5.35	26.75	53.5	107
1,4-Dichlorobenzene	0.107	0.214	0.535	1.07	5.35	26.75	53.5	107
sec-Butylbenzene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
p-Isopropyltoluene	0.101	0.202	0.505	1.01	5.05	25.25	50.5	101
1,2,3-Trimethylbenzene	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
1,2-Dichlorobenzene	0.104	0.208	0.520	1.04	5.20	26.00	52.0	104
d-Limonene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
1,2-Dibromo-3-Chloropropane	0.105	0.210	0.525	1.05	5.25	26.25	52.5	105
n-Undecane	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
1,2,4-Trichlorobenzene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
Naphthalene	0.095	0.190	0.475	0.95	4.75	23.75	47.5	95
n-Dodecane	0.102	0.204	0.510	1.02	5.10	25.50	51.0	102
Hexachlorobutadiene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
Methacrylonitrile	0.103	0.206	0.515	1.03	5.15	25.75	51.5	103
Cyclohexanone	0.096	0.192	0.480	0.96	4.80	24.00	48.0	96
tert-Butylbenzene	0.106	0.212	0.530	1.06	5.30	26.50	53.0	106
n-Butylbenzene	0.110	0.220	0.550	1.10	5.50	27.50	55.0	110

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 3A - Standard Concentrations (SIM) (Primary Sources)[1]

Compound Name	10pg	25pg	50pg	100pg	500pg	1000pg	2500pg	10,000pg	20,000pg
Freon-12	10.20	25.50	51.00	102.0	510	1020	2550	10200	20400
Chloromethane	9.90	24.75	49.50	99.0	495	990	2475	9900	19800
Vinyl Chloride	10.00	25.00	50.00	100.0	500	1000	2500	10000	20000
Bromomethane	10.00	25.00	50.00	100.0	500	1000	2500	10000	20000
Chloroethane	10.00	25.00	50.00	100.0	500	1000	2500	10000	20000
Acetone*	53.90	134.75	269.50	539.0	2695	5390	13475	53900	107800
Freon-11	10.20	25.50	51.00	102.0	510	1020	2550	10200	20400
1,1-Dichloroethene	10.90	27.25	54.50	109.0	545	1090	2725	10900	21800
Methylene Chloride	10.70	26.75	53.50	107.0	535	1070	2675	10700	21400
Freon-113	10.70	26.75	53.50	107.0	535	1070	2675	10700	21400
trans-1,2-Dichloroethene	10.50	26.25	52.50	105.0	525	1050	2625	10500	21000
1,1-Dichloroethane	10.40	26.00	52.00	104.0	520	1040	2600	10400	20800
Methyl tert-Butyl Ether*	10.60	26.50	53.00	106.0	530	1060	2650	10600	21200
cis-1,2-Dichloroethene	10.80	27.00	54.00	108.0	540	1080	2700	10800	21600
Chloroform	10.80	27.00	54.00	108.0	540	1080	2700	10800	21600
1,2-Dichloroethane	10.50	26.25	52.50	105.0	525	1050	2625	10500	21000
1,1,1-Trichloroethane	10.30	25.75	51.50	103.0	515	1030	2575	10300	20600
Benzene	11.00	27.50	55.00	110.0	550	1100	2750	11000	22000
Carbon Tetrachloride	11.00	27.50	55.00	110.0	550	1100	2750	11000	22000
1,2-Dichloropropane	10.30	25.75	51.50	103.0	515	1030	2575	10300	20600
Trichloroethene	10.30	25.75	51.50	103.0	515	1030	2575	10300	20600
Bromodichloromethane	10.60	26.50	53.00	106.0	530	1060	2650	10600	21200
1,4-Dioxane*	10.80	27.00	54.00	108.0	540	1080	2700	10800	21600
cis-1,3-Dichloropropene	9.90	24.75	49.50	99.0	495	990	2475	9900	19800
trans-1,3-Dichloropropene	11.10	27.75	55.50	111.0	555	1110	2775	11100	22200
1,1,2-Trichloroethane	10.20	25.50	51.00	102.0	510	1020	2550	10200	20400
Toluene	10.60	26.50	53.00	106.0	530	1060	2650	10600	21200
1,2-Dibromoethane	10.50	26.25	52.50	105.0	525	1050	2625	10500	21000
Tetrachloroethene	9.60	24.00	48.00	96.0	480	960	2400	9600	19200
Chlorobenzene	10.60	26.50	53.00	106.0	530	1060	2650	10600	21200
Ethylbenzene	10.40	26.00	52.00	104.0	520	1040	2600	10400	20800
m,p-Xylenes	20.40	51.00	102.00	204.0	1020	2040	5100	20400	40800
o-Xylene	10.10	25.25	50.50	101.0	505	1010	2525	10100	20200
1,1,2,2-Tetrachloroethane	9.80	24.50	49.00	98.0	490	980	2450	9800	19600
1,3-Dichlorobenzene	10.70	26.75	53.50	107.0	535	1070	2675	10700	21400
1,4-Dichlorobenzene	10.70	26.75	53.50	107.0	535	1070	2675	10700	21400
1,2-Dichlorobenzene	10.40	26.00	52.00	104.0	520	1040	2600	10400	20800
1,2-Dibromo-3-chloropropane	10.50	26.25	52.50	105.0	525	1050	2625	10500	21000
1,2,4-Trichlorobenzene	10.60	26.50	53.00	106.0	530	1060	2650	10600	21200
Naphthalene	9.50	23.75	47.50	95.0	475	950	2375	9500	19000
Hexachloro-1,3-butadiene*	10.60	26.50	53.00	106.0	530	1060	2650	10600	21200

*Reported upon request.

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 4 - Standard Concentrations (SCAN) (Secondary Sources)[1]

Compound Name	25ng	Compound Name	25ng	Compound Name	25ng
Bromochloromethane (IS1)	**25.0**	1,1,1-Trichloroethane	25.50	alpha-Pinene	24.00
Propene	25.50	Isopropyl acetate	55.00	n-Propylbenzene	24.75
Dichlorodifluoromethane (CFC 12)	25.25	1-Butanol	52.25	3-Ethyltoluene	25.50
Chloromethane	24.50	Benzene	26.00	4-Ethyltoluene	25.50
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	25.75	Carbon Tetrachloride	26.50	1,3,5-Trimethylbenzene	26.00
Vinyl Chloride	25.00	Cyclohexane	50.25	alpha-Methylstyrene	25.50
1,3-Butadiene	26.25	tert-Amyl Methyl Ether	25.75	2-Ethyltoluene	25.25
Bromomethane	25.00	1,2-Dichloropropane	25.50	1,2,4-Trimethylbenzene	25.00
Chloroethane	25.25	Bromodichloromethane	25.50	n-Decane	25.00
Ethanol	119.75	Trichloroethene	24.75	Benzyl Chloride	25.75
Acetonitrile	25.25	1,4-Dioxane	25.75	1,3-Dichlorobenzene	25.75
Acrolein	25.50	Isooctane	25.25	1,4-Dichlorobenzene	26.50
Acetone	129.50	Methyl Methacrylate	51.75	sec-Butylbenzene	26.00
Trichlorofluoromethane	26.25	n-Heptane	25.25	p-Isopropyltoluene	24.25
Isopropyl Alcohol	49.50	cis-1,3-Dichloropropene	24.50	1,2,3-Trimethylbenzene	26.00
Acrylonitrile	25.75	4-Methyl-2-Pentanone	26.25	1,2-Dichlorobenzene	25.50
1,1-Dichloroethene	27.25	trans-1,3-Dichloropropene	27.25	d-Limonene	25.75
tert-Butanol	51.75	1,1,2-Trichloroethane	25.25	1,2-Dibromo-3-Chloropropane	25.25
Methylene Chloride	26.50	**Chlorobenzene-d5 (IS3)**	25.0	n-Undecane	25.50
Allyl Chloride	26.75	**Toluene-d8 (S)**	**25.0**	1,2,4-Trichlorobenzene	25.00
Trichlorotrifluoroethane	26.50	Toluene	26.00	Naphthalene	22.25
Carbon Disulfide	26.00	2-Hexanone	28.50	n-Dodecane	22.75
trans-1,2-Dichloroethene	25.25	Dibromochloromethane	27.00	Hexachlorobutadiene	26.00
1,1-Dichloroethane	25.75	1,2-Dibromoethane	26.00	Methacrylonitrile	25.75
Methyl tert-Butyl Ether	25.50	Butyl Acetate	28.50	Cyclohexanone	23.75
Vinyl Acetate	123.50	n-Octane	25.75	tert-Butylbenzene	26.50
2-Butanone (MEK)	26.50	Tetrachloroethene	23.75	n-Butylbenzene	26.50
cis-1,2-Dichloroethene	26.75	Chlorobenzene	26.00		
Diisopropyl Ether	26.50	Ethylbenzene	25.75		
Ethyl Acetate	51.50	m- & p-Xylene	51.50		
n-Hexane	25.75	Bromoform	27.00		
Chloroform	27.75	Styrene	26.00		
1,2-Dichloroethane-d4 (S)	**25.0**	o-Xylene	25.00		
Tetrahydrofuran	26.00	n-Nonane	25.25		
Ethyl tert-Butyl Ether	25.50	1,1,2,2-Tetrachloroethane	24.75		
1,2-Dichloroethane	26.00	**4-Bromofluorobenzene (S)**	**25.0**		
1,4-Difluorobenzene(IS2)	**25.0**	Cumene	24.50		

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 4A – ICV/LCS Standard Concentrations (SIM) (Secondary Sources)[1]

Compound Name	500pg
Freon-12	505
Chloromethane	490
Vinyl Chloride	500
Bromomethane	500
Chloroethane	505
Acetone*	2590
Freon-11	525
1,1-Dichloroethene	545
Methylene Chloride	530
Freon-113	530
trans-1,2-Dichloroethene	505
1,1-Dichloroethane	515
Methyl tert-Butyl Ether*	510
cis-1,2-Dichloroethene	535
Chloroform	555
1,2-Dichloroethane	520
1,1,1-Trichloroethane	510
Benzene	520
Carbon Tetrachloride	530
1,2-Dichloropropane	510
Trichloroethene	495
Bromodichloromethane	510
1,4-Dioxane*	515
cis-1,3-Dichloropropene	490
trans-1,3-Dichloropropene	545
1,1,2-Trichloroethane	505
Toluene	520
1,2-Dibromoethane	520
Tetrachloroethene	475
Chlorobenzene	520
Ethylbenzene	515
m,p-Xylenes	1030
o-Xylene	500
1,1,2,2-Tetrachloroethane	495
1,3-Dichlorobenzene	515
1,4-Dichlorobenzene	530
1,2-Dichlorobenzene	510
1,2-Dibromo-3-chloropropane	505
1,2,4-Trichlorobenzene	500
Naphthalene	445
Hexachloro-1,3-butadiene*	520

*Report upon request

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

ATTACHMENT A

Training Plan for Analysis of VOCs by GC/MS

Training Plan for Analysis of VOCs by GC/MS

Trainee ________________ Trainer _________________ Instrument _____ Training Completion Date _____________

1. Read SOP *Training Duration* ______________ Trainer ___ Trainee ___ Date ___________

2. Read Methods TO-14A & TO-15A *Training Duration* ______________ Trainer ___ Trainee ___ Date ___________

3. Demonstrated understanding of the scientific basis of the analysis Trainer ___ Trainee ___ Date ___________
   Whole air sample preconcentration techniques
   Gas chromatography *Training Duration* ___________________
   Mass spectrometry

4. Demonstrated familiarity with related SOPs Trainer ___ Trainee ___ Date ___________
   SOP for Batches and Sequences; Rev. ___
   SOP for Making Entries onto Analytical Records; Rev. ___ *Training Duration* ___________________
   SOP for Manual Integration Policy; Rev. ____
   SOP for Significant Figures; Rev. ____
   SOP for Nonconformance and Corrective Action; Rev. ____
   SOP for Performing MDL Studies and Establishing Limits of Detection and Quantitation; Rev. ____
   SOP for Cleaning and Certification of Summa Canisters; Rev. ___

5. Observe performance of SOP *Training Duration* ______________ Trainer ___ Trainee ___ Date ___________
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant introduction (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)

6. Perform SOP with supervision *Training Duration* ______________ Trainer ___ Trainee ___ Date ___________
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant use (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)

7. Independent performance of the SOP *Training Duration* ______________ Trainer ___ Trainee ___ Date ___________
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant proficiency (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)
   ___initial demonstration of competency (4 Laboratory Control Samples)

8. Instrument operation and maintenance Trainer ___ Trainee ___ Date ___________
   ___autosampler *Training Duration* ___________________
   ___GC and capillary column installation *Training Duration* ___________________
   ___mass spectrometer *Training Duration* ___________________
   ___data system *Training Duration* ___________________

ATTACHMENT B

Initial Calibration Review Checklist

Initial Calibration Review Checklist - EPA Compendium Method TO-15

ICAL Date:______________________ ICAL ID:____________________ LIMS ICAL ID:____________________

Instrument: ☐ MS3 ☐ MS7 ☐ MS8 ☐ MS9 ☐ MS11 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21

Mode: ☐ SIM ☐ Scan Scan Low Level (0.1ng): ☐ Yes ☐ No

Analyst **Reviewer**

☐ 1. Is the required documentation in the ICAL file? .... ☐
- ☐ BFB Tune analysis Report .... ☐
- ☐ Calibration Status Report (aka Calibration History) .... ☐
- ☐ Response Factor Report/Percent RSD .... ☐
- ☐ Quantitation Report for each calibration standard (including manual integration documentation)....... ☐
- ☐ ICV Quantitation Report .... ☐
- ☐ TO-15 Standard Calculation Spreadsheet .... ☐

☐ 2. Was the ICAL performed continuously (not interrupted for maintenance or sample analysis)? .... ☐

☐ 3. Have all the calibration standards been analyzed within 24 hours of each other? .... ☐

☐ 4. Does the BFB tune check standard analysis at the start meet the tune criteria? .... ☐

☐ 5. Are all the analytes in the blank analysis <MRL? .... ☐

☐ 6. Does each analyte's ICAL include a minimum of 5 concentrations at 5 consecutive levels? .... ☐

☐ 7. Were the standards analyzed from low concentration to high concentration? .... ☐

☐ 8. For each analyte, are there no levels skipped? .... ☐

☐ 9. For each analyte, is there only one value used for each calibration level? .... ☐

☐ 10. For each analyte, is the lowest standard's concentration at or below the analyte's MRL? .... ☐

☐ 11. For each analyte, is the corresponding signal to noise ratio at least 3:1 at the lowest point on the curve? .... ☐

☐ 12. For each analyte, are the corresponding upper levels free from saturation? .... ☐

☐ 13. If a calibration level is dropped, are all the responses for each target analyte dropped and is the information noted in the ICAL explaining the reason? .... ☐

☐ 14. Is the average RSD ≤30% for all analytes, with no more than two exceptions ≤40%? .... ☐

☐ 15. Is the response Y at each calibration level within 40% of the mean area response over the initial calibration range for each internal standard? .... ☐

☐ 16. Percent recovery for each analyte in the ICV 70%-130% (50-150% for VA, unless AFCEE or DoD)? .... ☐

☐ 17. Was the RRT for each target compound at each calibration level within 0.06RRT units of the mean RRT for the compound? .... ☐

☐ 18. Is the retention time shift for each of the internal standards at each calibration level within 20s of the mean retention time over the initial calibration range for each standard? .... ☐

☐ 19. If there are any manual integrations, are they performed correctly according to the corresponding SOP? If so, initial and date the appropriate pages. .... ☐

☐ 20. Is the ICAL good at 0.5ng (or 0.1ng) – 100ng (Scan) or 10-20000pg (SIM) for all compounds? ☐ Yes ☐ No
If not, note exceptions and the corresponding MRLs below – *Specify applicable range...* .... ☐

☐ 21. Are ALL of the peak selections for each analyte correct according to retention time (all RTs must be checked by both the initial and peer reviewer)? .... ☐

COMMENTS:

Analyst:______________________________ Date: ______________________

Secondary Reviewer: ______________________ Date: ______________________

ATTACHMENT C

Data Review Checklist

EPA Compendium Method TO-15 - Data Review Checklist

(Note exceptions in Comments Section and attach Analysis Observations / Case Narrative Summary Form as appropriate)

Method: ☐ EPA TO-15 ☐ EPA TO-14A **Analysis Date:** ____________________ **Project #:** ____________________

Instrument: ☐ MS3 ☐ MS7 ☐ MS8 ☐ MS9 ☐ MS11 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21

Mode: ☐ SIM ☐ Scan **Scan Low Level (0.1ng):** ☐ Yes ☐ No **DOD:** ☐ Yes ☐ No

Analyst **Reviewer**

☐ 1. Is the required documentation present? .... ☐
- ☐ CORRECT BFB Tune analysis Report
- ☐ CCV analysis Quantitation Report & %D Report
- ☐ Duplicate analysis Quantitation Report
- ☐ Quantitation Report for each sample included
- ☐ Manual Integration & Q Deletion Documentation
- ☐ LCS analysis Quantitation Report
- ☐ MB analysis Quantitation Report
- ☐ Spectral match details for each hit, where necessary

☐ 2. Does the BFB **tune** check standard analysis meet the tune criteria for the method indicated above? .... ☐

☐ 3. Are all analyses within the tune's **24-hr window or** ☐ **Client's 12hr window requirement**? .... ☐

☐ 4. Does the **CCV** have a difference ≤30% for all analytes? .... ☐
[Note <u>all</u> outliers biased high and/or low]

☐ 5. Are all the **IS** retention times within 20 seconds of the CCV RT or the RT from the midpoint (ICAL)? .... ☐

☐ 6. Are all the **IS** responses within ±40% of CCV or the midpoint in the ICAL? .... ☐

☐ 7. Are all the **surrogate** recoveries (in CCVs, MB, LCSs, etc.) within acceptance limits (70%-130%) .... ☐

☐ 8. Are all the analytes in the **MB** <MRL? (DoD <1/2MRL, except Acetone, MeCl2, EtOH, Carbon Disulfide)? .... ☐

☐ 9. Is the **LCS** %R within the lab control limits for all analytes except AZ samples (70%-130%, VA 50%-150%)? . ☐

☐ 10. Are all the analytes in the **Lab Duplicate / DLCS** within ±25% or the client specified limits? .... ☐

☐ 11. Are all the analyte hits in the samples within the **calibration range** and/or noted? .... ☐

☐ 12. Are all **peak integrations** acceptable? .... ☐

☐ 13. Are all **manual integrations** flagged and documented? .... ☐

☐ 14. Are ALL of the peak selections for each analyte correct according to **retention time**? .... ☐

☐ 15. Are all the Q Deletions correct? .... ☐

☐ 16. Are all **calculations** correct? .... ☐

☐ 17. Has the analyst initialed and dated each **quantitation report**? .... ☐

☐ 18. For **TICs** are the relative intensity and other requirements met? .... ☐

☐ 19. **Auto report** correct? .... ☐

☐ 20. **MRL** = _______ ☐ ug/m3 ☐ pg (ethanol, acetone, vinyl acetate = 5.0ug/m3) .... ☐

☐ 21. Pressurized with **Helium**? Is the worksheet completed for all samples? .... ☐

☐ 22. Report to **MDL**? ☐ Yes ☐ No .... ☐

☐ 23. **Global Minimum Detection Limit** = _______ ☐ ug/m3 ☐ pg .... ☐

☐ 24. **DOD:** Are **manual integrations** notated in the **case narrative?** .... ☐

COMMENTS:

☐ LIMS Run Approval ☐ LIMS Supervisor Approval

Analyst: ______________________________ Secondary Reviewer: ______________________________

Date: ______________________________ Date: ______________________________

ATTACHMENT D

State and Project Specific Requirements

Minnesota Requirements	
Item	**Criteria**
Holding Time (HT)	14 days
Tedlar bags	Not allowed for sampling or sample dilution
Canisters and flow controllers	Individually certified Individually leak checked before shipment
	Samples with concentrations outside of the calibration curve will have a zero canister analysis performed to check for carryover. If carryover is detected, system bake out shall be performed and documented. Additionally, in instances where the laboratory has evidence on file that a particular compound when present at a high concentration does not exhibit carry-over, the samples will not be reanalyzed. When samples are analyzed that have a higher concentration than the evidence on file, the above requirements must be followed. Also, samples that have hits below the MRL will not be reanalyzed when analyzed after a sample with concentrations over the calibration range.
Method Reporting Verification Check	Analyze a Method Reporting Verification at the beginning of the sequence prior to analyzing samples. Acceptance criteria ±40%.
Duplicates	10 percent laboratory duplicates
Record retention	MN/NELAC 5 years MPCA (Minnesota Pollution Control Agency) compliant samples 10 years
Tier level	TIII

Arizona Requirements	
Item	**Criteria**
LCS	70-130% (vinyl acetate 50-150%)

Department of Toxic Substances Control (DTSC) Requirements	
Item	**Criteria**
Holding Time (HT)	72 hour hold time for canisters

EPA Region 9 Requirements	
Item	**Criteria**
Holding Time (HT)	14 days

Attachment D
Responses to Agency Comments on Draft Work Plan

Attachment D—Response to Comments

This attachment presents responses to comments from the US Environmental Protection (USEPA) and New Jersey Department of Environmental Protection (NJDEP) on the Draft Work Plan for the Winter 2013/2014 Monitoring Events at 115 River Road, 163 Old River Road, and 103 River Road at the Quanta Resources Corporation Superfund Site, Operable Unit 1 (OU1).

Comments were received via e-mail from Richard Ho/USEPA on November 5, 2013. Responses to the comments received November 5 were drafted and submitted to USEPA via e-mail on November 20, 2013. On November 26, 2013, an e-mail was received with three additional comments from NJDEP that were based on the responses to the November 5 comments. There were no comments on the proposed plan for vapor intrusion (VI) monitoring at the 163 Old River Road, and 103 River Road buildings.

Comments Received November 5, 2013

Comment 1:

As stated in the work plan, a number of sample locations have been modified from previous sampling rounds. Indoor air samples are not proposed for building 3, the upper floors of Buildings 7, 9 and 10, and the lower floors of Buildings 8 (1st floor) and 10 (basement).

Based on discussions at the recent meeting NJDEP had with the RP and EPA, the omission of samples from these locations may be because these areas of the building are not currently in use. This needs to be verified. The omission of samples in an area that is not used must include verification of the cited lack of use along with monitoring to ensure that use of the subject area does not change in the future.

Response to Comment (RTC) 1:

The scope of the annual VI monitoring at the 115 River Road building includes indoor air sampling in each of the occupied first floor tenant spaces, and discontinues indoor air sampling in unoccupied tenant spaces. Indoor air sampling is generally not proposed for the upper floors of the 115 River Road Building because historical sampling results from 2006 to 2013 indicate that the VI pathway is not complete and significant (i.e., resulting in indoor air concentrations above regulatory target levels) at the building. Indoor air sampling was performed in some of the upper floors in 2006 (e.g., Buildings 3, 8, 9, and 10), and then was discontinued after that in accordance with the agency approved monitoring work plans from 2008 to 2013. Indoor air samples will be collected from the second and third floor occupied tenant spaces closest to the elevator in Building 8 to assess if the elevator is acting as a preferential vapor pathway as discussed in the work plan.

The occupancy status of each tenant space in the building was provided by the building owner in August 2013. Since then CH2M HILL has checked for changes in occupancy through communication with the building manager during bi-monthly site inspections. The occupancy conditions documented during the November 7 site inspection do not change the proposed sampling plan for the Winter 2013/2014 VI Monitoring Event.

Explanations of proposed sampling in specific areas of the building are provided below:

- Indoor air sampling is not proposed for Building 3 because the first floor is unoccupied.
- Buildings 7 and 8 are combined in such a way that there are no first floor tenant spaces in Building 8 (there is only the elevator lobby), and there are no third floor tenant spaces in Building 7 (the third floor of both buildings is considered to be Building 8). Indoor air sampling is not proposed for the second floor of Building 7 because historical indoor air sampling results have indicated that there is limited spatial variability (i.e., less than 1.5-times) between the Building 7 first and second floor sample results.
- There is only one tenant space in Building 9, the Osteo Relief Clinic, which has an “open” design (i.e., the majority of the tenant space, including the second floor, is one large connected indoor air volume). Indoor air samples will be collected from the two previously sampled first floor locations in Building 9.

- The work plan will be revised to include one indoor air sample in the Building 10 basement as discussed below in RTC 2.

Comment 2:

The sub-slab soil gas sample (VI-10) collected from building 9 and indoor air sample (IA-22) collected near the sumps in building 10 have also been eliminated. It should be noted that absence of sub slab or indoor air samples from the lower floors will eliminate use of these data as part of the multiple lines of evidence approach in evaluating background sources to indoor air in relation to VI.

RTC 2:

The Building 9 subslab soil gas probe Q1-VI-10 was sampled in 2010, 2011, and 2013. The results have been mostly non-detects, with no detected VOC concentrations above regulatory screening levels. The measured subslab soil gas concentrations do not correlate with those in indoor air. The measured VOC concentrations in subslab soil gas are less than those in indoor air; for example, the measured subslab soil gas benzene concentration in March 2013 was 0.68 $\mu g/m^3$ compared to 3.2 and 5.9 $\mu g/m^3$ in the two first floor indoor air samples. Therefore, the subslab soil gas sample results from Q1-VI-10 are not a valuable line of evidence in evaluation of VI at Building 9.

The work plan will be revised to include one indoor air sample in the Building 10 basement at the previously sampled location, Q1-IA-22, because this data will be useful as a line of evidence when evaluating the first floor indoor air sample results.

Comments Received November 26, 2013

Comment 1:

No sampling is proposed in Building 3. The submission indicates the first floor is unoccupied but does not mention if the upper floor is.

The data indicates that the only indoor air sample for the second floor was collected in 2006. If the second floor is still occupied, DEP recommends that one indoor air sample be collected from the second floor occupied space.

RTC 1:

The second floor of Building 3 is occupied. Indoor air sampling was performed in the second floor of the building tenant space in March and July 2006 and was then discontinued in accordance with the agency-approved VI monitoring work plans from 2008 through 2013. Multiple lines of evidence, including the NAPL plume boundary, shallow groundwater volatile organic compound (VOC) concentrations, and indoor and crawl space air sample results from 2006 to 2013, indicate that the VI pathway is not complete or significant at Building 3. Therefore, indoor air sampling in the second floor tenant space at Building 3 in not proposed for the winter 2013–2014 VI monitoring event.

In response to additional comments received from EPA via email on December 13, 2013, one indoor air sample will be collected in the Building 3 second floor tenant space and one sample will be collected in the Building 3 crawl space as additional line of evidence for the VI pathway.

Comment 2:

DEP recommends that an indoor air sample should be collected in an occupied area of Building 7 (second floor) where previous exceedences of the NJDEP screening levels have been detected. The data indicates that second floor sample (IA-38) had an elevated benzene level (2.9 $\mu g/m^3$) during the March 2013 sampling event. This location should be resampled.

RTC 2:

Indoor air samples will be collected from two locations in the first floor of Building 7. Indoor air sampling is not proposed for the second floor of Building 7 because historical indoor air sampling results have indicated that there is limited spatial variability (i.e., less than 1.5 times) between the Building 7 first and second floor sample results. The measured indoor air benzene concentrations at the two first floor sample locations (Q1-IA-36 and Q1-IA-37) in March 2013 were higher (4.6 and 3.1 μg/m^3) than the measured indoor air benzene concentration at the second floor location (Q1-IA-38; 2.9 μg/m^3). This same pattern was observed for the majority of the other analytes and is conceptually valid because the first floor is closer to the basement. Therefore, sample results from the two first-floor locations are more representative of a “worst-case” scenario than sample results from the second floor location.

Comment 3:

The Building 9 first floor indoor air samples had previous exceedences of the Department’s indoor air screening levels for benzene, ethyl benzene and naphthalene. The response is unclear but appears to indicate the second floor is occupied and has an “open” design. DEP recommends an indoor air sample be collected from the second floor, if there is an occupied space that is enclosed and not part of the large connected open design area.

RTC 3:

Indoor air samples will be collected from two locations in the first floor of Building 9. Indoor air sampling is not proposed for the second floor of Building 9 because of an “open” design and a heating, ventilation, and air condition system that results in one large, connected indoor air volume. Although there are several offices on the second floor of Building 9, the doors are most often kept open. Furthermore, the two first-floor sample locations are representative of “worst-case” VI locations, especially the east side storage room location (Q1-IA-41) that is above the Building 7/8 basement and where odors have been observed. Therefore, sample results from the two first-floor locations are more of a worst case, than sampling from a second-floor location.